

557
IL6of
1987-2

Geol Survey

Final Technical Report

THE ISGS AGGREGATE FLOTATION FINE COAL CLEANING PROCESS

PART A: Development, Characterization and Testing

Illinois State Geological Survey

Open file series 1987-2

Submitted to the
Illinois Coal Development Board
through the
Center for Research on Sulfur in Coal

August 1987

ILLINOIS STATE GEOLOGICAL SURVEY



3 3051 00007 2953

FINAL TECHNICAL REPORT
September 1, 1986 through August 31, 1987

Project Title: THE ISGS AGGREGATE FLOTATION FINE COAL CLEANING PROCESS
PART A: DEVELOPMENT, CHARACTERIZATION AND TESTING

Principal Investigators: R. B. Read and P. J. DeMaris, ISGS

Co-Investigators: D. M. Rapp, L. R. Camp, M. S. Summers, ISGS
and C. Y. Meyers, SIUC

ABSTRACT

The Illinois State Geological Survey, in collaboration with Northwestern University and Southern Illinois University, has been developing a physical ultrafine coal cleaning process, Aggregate Flotation (AF). The major objective is to significantly reduce pyritic sulfur and ash-bearing minerals from Illinois Basin coals while maintaining high levels of Btu recovery. Rejections of 80% to 90% ash and pyritic sulfur can be achieved by the AF process while Btu recoveries are generally maintained at >80% (ROM basis). Technological advances in "chemical control" of the ultrafine coal flotation process reduces the need to develop or utilize new types of cells for producing ultrafine bubbles.

Results using a number of proprietary reagents for three Illinois Basin Coals have been determined. For a prewashed Illinois No. 5 (Springfield-SE) coal an additional 45% to 50% rejection of ash and pyrite at >80% Btu recovery can be attained if the preparation plant product is treated by the AF process. For a Run-of-Mine (ROM) No. 5 coal, 70% to 80% ash and pyrite rejection at 90% Btu recovery were obtained. Similar results for a difficult-to-float, ROM Illinois No. 2 (Colchester-W) coal showed ash and pyrite rejections on the order of 60% at the 80% Btu level. New synthetic reagents developed at SIU have been shown to be extremely effective in reducing the dosage required for effective flotation of both ultrafine (-400 mesh) and conventional size flotation feeds (28x0). Synergism of alcohol/surfactant mixtures has been shown for several feed coals. Additional studies on the synergistic effects for other Illinois No. 6 (Herrin-SC1, SC2) coals are in progress.

Recent batch tests on a precleaned Illinois No. 6 (Herrin-SC2) coal indicate that compliance levels ($1.2\text{#SO}_2/\text{MMBtu}$) are attainable at 92% to 95% Btu recovery. Additional results for this high chlorine coal indicate ultrafine grinding reduced the ROM chlorine content from 0.35 wt % to 0.15 wt %, a reduction of approximately 60%.

Results obtained from a comparative study between ISGS and United Coal Company (UCC) on the Illinois No. 5 (Springfield-SE) coal indicate AF is a superior method when contrasted to column flotation for reduction of ash and sulfur. The AF process also yielded ultraclean (<1% ash, <1% total sulfur) coal from samples (Cedar Grove Seam, VA) provided by UCC.

Characterization of petrographic and mineralogic constituents has permitted better interpretation of the degree to which pyritic sulfur and ash can be removed from various coals. A new petrographic technique, "mineral liberation analysis" was developed which yields more direct information about the liberation potential of the remaining minerals in the processed samples.

EXECUTIVE SUMMARY

Over the course of the past four years, the Minerals Engineering Section at the Illinois State Geological Survey (ISGS), in conjunction with support from Northwestern University (NU) and Southern Illinois University (SIU), has been developing an ultrafine, (-400 mesh) advanced physical coal cleaning process - ISGS Aggregate Flotation (AF). The basic process incorporates aspects from both oil agglomeration and froth flotation. Extensive chemical, petrographic and mineralogical support has aided in delineating appropriate process parameters. The combined effort has led to a potentially viable option for significant reduction of pyrite and ash-forming minerals in bituminous coals. A brief synopsis is given below.

- * The AF process will generally retain over 80% Btu recovery while rejecting over 80% of the pyritic sulfur and ash in run-of-mine coal.
- * For prewashed coals, an additional 40% to 50% reduction in ash and pyrite is obtained while maintaining over 80% Btu recovery.
- * Production of ultra-clean coal (<1% ash, <1% total sulfur) was demonstrated with the AF process for the Cedar Grove (VA) coal.
- * Conventional froth flotation equipment may be utilized in the AF process. The unique character of ultrafine microbubbles is controlled by specific, proprietary reagents and not equipment.
- * The AF process has shown improved cost effectiveness regarding Btu recovery, ash and pyrite rejection when compared to conventional processes such as froth flotation or oil agglomeration.
- * Use of proprietary surfactants, developed at SIU and tested in the AF process, has reduced the required dosage to produce equivalent Btu recovery by a factor of three to seven-fold as compared to conventional alcohol based frothers.
- * AF is successful in recovering saleable product from waste streams and gob piles. Preliminary results on waste stream feeds show high Btu recovery (80%) with reasonable ash rejection. A pre-AF step, such as tabling, may be required for gob.
- * Coal Petrography has been found to be a useful tool in accessing the size and association of pyrite grains in feed and various product samples. Trends in mean grain diameter and Pyrite Cleanability Indices which correlate with percent pyritic sulfur values, have clarified changes occurring in both grinding and cleaned sample sets.
- * A new petrographic method "Liberation Analysis" has been developed which more comprehensively characterizes major minerals (including pyrite) remaining in the finely ground samples.

The technical objective of the process is to develop an economical approach for pyritic sulfur and ash reduction from difficult-to-clean Illinois Basin bituminous coals. Examination of other coals, including those from

Appalachia, have indicated the process is applicable to coals from other regions as well.

Major advances in new physical coal cleaning technologies which address concerns regarding pending acid rain legislation are beginning to emerge. Not only are SO₂ emissions of concern, but benefits to coal burning utilities which accrue from removal of mineral matter and chlorine are receiving renewed interest. While it is recognized that physical coal cleaning methods will not remove organic sulfur, such methods have been shown to be advantageous in reducing requirements for desulfurization by processes such as limestone injection, fluidized bed combustion or stack gas scrubbing.

Aggregate Flotation (AF), a process which utilizes conventional froth flotation equipment and ultrafine grinding technology to liberate pyrite and mineral matter from coal, has been shown to be quite effective. Restrictions in achieving effective separation of ash and pyrite from ultrafine coal have been overcome by utilizing "chemically selective" reagents. By taking advantage of differences in surface properties of coal, mineral matter and pyrite, and applying principles based on coal surface chemistry/reagent composition to solve the problem of inefficient separations, extremely positive results have been attained. Results to date indicate substantial potential exists for removing the highly disseminated pyrite in many coals by grinding until apparent optimum pyrite liberation occurs, as indicated from petrography, and then selectively floating coal with surfactants, or surfactant/alcohol mixtures which promote the stabilization of small bubbles and the formation of coal-air aggregates. The initial economic evaluation appears encouraging.

Efforts in reagent development, obtained from a joint ISGS/SIUC study have achieved extremely positive results. A series of ten reagents of known chemical structure, prepared or obtained commercially, were evaluated for effectiveness in coal cleaning/pyrite removal by Aggregate (ISGS) and froth flotation processes. The series of new reagents were prepared by a method invented at SIUC some years ago and patented (assignments to the SIUC Foundation). A correlation was made between the structure of these reagents and their coal cleaning effectiveness and efficiency, i.e., dosage vs. yield, Btu recovery, ash and pyrite rejection, on an Illinois No. 5 (Springfield-SE) coal (hva) and an Illinois No. 2 (Colchester-W) coal (hvc). In the Aggregate Flotation studies, within a series of nine structurally related reagents, molecular size and geometry were found to be quite influential in determining coal cleaning ability. In general, the synthesized reagents were more effective than the commercial reagents. In some cases, the required dosage of the synthetic reagents was only one-third that of the commercial reagents and one-seventh that of the alcohol-based frother MIBC to produce equivalent Btu recoveries. Mixtures of the synthetic reagents and frothing alcohols were synergistically effective, producing higher Btu recovery and better ash/pyrite rejection than either reagent alone. The new synthetic reagents have been also shown to be effective for conventional size froth flotation feed (28x0) in the Upper Freeport coal.

Further efforts at optimizing reagent compositions used in the AF process have been undertaken for the IBCSP #4 (Illinois No. 6, Herrin-SW) sample.

Two other Illinois No. 6 samples, the Herrin SC-1 and SC-2, are currently undergoing similar evaluation. Results from the IBCSP #4 (Illinois No. 6, Herrin-SW) sample indicate it is significantly more difficult to float than the Illinois No. 5 (Springfield-SE) sample, but not as difficult to float as the Illinois No. 2 (Colchester-W) coal. High Btu recoveries for the IBCSP #4 (Illinois No. 6, Herrin-SW) were maintained (>80%) while rejecting over 80% of the ash and 65% of the pyritic sulfur (ROM basis). A series of Aggregate Flotation tests which compare rougher only and rougher/cleaner stages have also been completed for the Illinois No. 5 (Springfield-SE) ROM and tabled samples. There appears to be an overall improvement, at equivalent Btu recovery, of approximately 5%-8% in both ash and pyrite rejection when using a rougher/cleaner cycle for ROM coal, and a similar trend but with a lower degree of ash and pyrite rejection for the tabled product (3%-5%). The concentrate grades show a slight improvement with the rougher/cleaner cycle.

In related studies, characterization of coal surface properties and their interactions with reagents used in the AF process have been initiated by measuring the zeta potential of two coals. Measurements of whole coals both in water and with surfactants showed the zero point of charge (zpc) to be in the range of 4 to 4.5 for the Illinois No. 5 (Springfield-SE) sample, but the Illinois No. 2 (Colchester-W) sample had a zpc of pH=6, an unexpectedly high value. Two non-ionic surfactants exhibited relatively little influence on the potential determining ions, whereas the zeta potential for the anionic surfactant was shown to be significantly reduced (negative). This finding may indicate that the nonionic reagents control surface tension, whereas the presence and usage of charged anionic surfactants imply adsorption onto the surfaces of the coal particles.

Comparison of AF and United Coal Company (UCC) column flotation techniques (microbubble) has been completed for the Illinois No. 5 (Springfield-SE) coal. For the single stage tests with near equivalent recovery (85%), the AF process shows a significantly superior degree of ash rejection (Column = 49.5%, AF = 87.3%) with better total sulfur rejection and lower product ash and total sulfur. For the dual stage tests, results are difficult to compare at equal recovery due to low values obtained by column flotation (46%). Nevertheless it is quite obvious that the AF process yields a much higher Btu recovery (almost two fold greater) and with higher ash and total sulfur rejection when compared to column flotation. These results suggest that AF is the superior method for cleaning the ultrafine Illinois No. 5 (Springfield-SE) coal. The AF process was also applied to a Virginia coal (Cedar Grove Seam) which UCC is currently utilizing for production of ultraclean coal (< 1% ash, total sulfur). With AF, a final product of 0.96% ash and 0.54% total sulfur was obtained at 90% Btu recovery.

Samples of waste from a washing plant of a major Illinois coal producer were studied in the laboratory. It was found that by screening and tabling, a saleable grade (+100 mesh) of coal was obtained. Treatment of the -100 mesh fraction of the plant outwash by AF yielded a 94% Btu recovery while rejecting 90.1% of the ash and 70.4% of the pyritic sulfur.

Characterization of products and waste by chemical, petrographic and XRD are aiding in the interpretation of the liberation/flotation phenomena.

Quantification of the liberation of ash and pyritic sulfur as a function of particle size has been initiated to determine the economic balance between grinding and rejection values obtained in fine (80% passing 65 mesh to 80% passing 200 mesh) versus ultrafine (80% passing 400 mesh) coal. Flotation results for differences in degree of rejection as a function of particle size will be compared in the upcoming year to those obtained previously for ultrafine grinding. Characterization by petrography, XRD and chemical analyses for various degrees of mineral matter liberation are initially reported and discussed in conjunction with the petrographic study. In particular, more emphasis in the future will be placed on ash compositional studies for ROM, gravity and AF products, which may provide key information on such products when utilized as a feedstock for combustion.

Based on the results obtained to date, ISGS Aggregate Flotation appears to be an extremely effective ultrafine, physical coal cleaning process. It is a technically viable approach for significantly reducing the ash and pyritic sulfur contents of Illinois Basin coals and can be utilized for a number of different feedstocks including ROM, prep plant products, waste tailings and gob. Ultrafine grinding, which achieves liberation of ash-bearing minerals, has been integrated with the use of "chemically selective" reagents which are specific for flotation of ultrafine coal. In this respect the need for developing or utilizing new types of cells (eg. columns, microbubble) has been avoided. Success in reagent development again, has been extremely promising.

The goal for the next year will be to continue both the fundamental and advanced development of the Aggregate Flotation process. Areas which may lead to decreased cost or technical improvements in the process will be sought. For example, defining the overall economic limit for the fine grinding/liberation/flotation process may be accomplished by studying different particle size feed coals as a function of Btu recovery, ash and pyrite rejection. Thus one could identify the cost/benefit ratios for fine (200 mesh) versus ultrafine (400 mesh) feed. Incorporated in this effort will be studies for effects of grinding on chlorine removal. Such issues as economics of operation, generating accurate, reliable data from the CFU for process evaluation, and further reagent composition optimization will be undertaken.

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	1
EXECUTIVE SUMMARY	2
TABLE OF CONTENTS	6
OBJECTIVES	7
INTRODUCTION	8
EXPERIMENTAL	10
AF Flotation Procedure	11
Experimental Approach Used to Attain Objectives	11
RESULTS AND DISCUSSION FOR BASIC AND APPLIED AF STUDIES	12
Comparison of Surfactant Efficiency Based on Structure, Type and Cost Effectiveness	12
Comparison of Collector and Surfactant/Collector Efficiency Based on Structure and Type	13
Kinetic Effects by Specific Surfactants	19
Electrokinetic Properties of Coals Used in the AF Process	23
Effectiveness of ISGS Aggregate Flotation as Compared to Alternate Microbubble Processes	26
Effectiveness of ISGS Aggregate Flotation as Compared to UCC Column (Microbubble) Flotation	29
Production of Ultraclean Coal With <2% Ash by ISGS Aggregate Flotation	30
Comparison of Reduction of Ash and Sulfur in Illinois Coals by UCC Column Flotation	30
Characterization of the Mineral Matter-Liberation Phenomena	34
Production of Compliance (1.2#SO ₂ /MMBtu) Illinois Basin Coal	34
Influence of Circuitry Design	37
Recovery of Coal From Prep Plant Waste Streams	39
Options for Aggregate Flotation Product Utilization	40
Coal-Water Slurry Option	41
Pelletization or Briquetting Option	41
Preliminary Combustion Tests	42
RESULTS AND DISCUSSION OF PETROGRAPHY AND XRD STUDIES	43
Sample Characterization	43
Evaluation of Liberation Data	44
Evaluation of Products From Waste Slurries	50
Mineralogic Studies	52
Future Petrographic Work	52
CONCLUSIONS AND FUTURE EFFORTS	53
ACKNOWLEDGEMENTS	54
REFERENCES	55
APPENDIX A	58

OBJECTIVES

The overall, long term objectives of this project have been to develop, refine, and prepare for eventual commercial testing, a promising physical fine coal cleaning process - Aggregate Flotation. Several years of intensive research and batch-testing have repeatably confirmed it to be a viable candidate for possible commercial utilization. Specific objectives for this 1986-1987 funding period include the following tasks.

Conduct major refinement and batch-testing of the AF process. This involved many aspects which included:

- * Critical comparison of surfactant efficacy based on structure, type and cost effectiveness.
- * Critical comparison of surfactant/collector efficacy based on structure, type, and cost effectiveness.
- * To employ best surfactant/collector combinations as determined on a batch basis in the continuous flow unit.
- * Comparison of the effectiveness of the ISGS AF process versus other known microbubble processes which utilize new cell designs.
- * Examination of options for final product form.
- * Conduct appropriate electrokinetic surface studies in order to better understand surfactant applicability in coal cleaning.
- * Where feasible, demonstrate that compliance coal can be obtained by the AF process.
- * Demonstrate AF feasibility on coal waste streams and refuse materials.

Continue progress toward eventual commercial testing of the AF process. This involved review of feasibility and planning, and was experimentally approached as follows:

- * Employing standardized instrument conditions and equivalent surfactant dosages (on a lb active surfactant/ton dry feed basis) resulting product quality and recovery were evaluated. This investigation yielded data which allowed facile comparison of different surfactants based on structure, type, and cost-effectiveness.
- * Using a "kinetic" approach, floated, timed fractions for a given surfactant dosage yielded information such as "cut-point" criteria based on Btu recovery, ash and pyrite rejection.
- * After selection of the most appropriate surfactant, a collector/surfactant match was attempted. Again, standard instrument con-

ditions and a statistical approach to surfactant/collector dosages (i.e., interactions) were employed.

- * Based on a final cost effective surfactant/collector pair(s), usage in the continuous plant was expected to yield cost and feasibility estimates for the process.
- * Determine level of upgrading required in the existing continuous flow unit and obtain needed equipment to insure successful operation.
- * Propose, promote, and participate in pilot stage testing of the AF process where appropriate and available.
- * Depending upon availability of pilot-plant facilities and successful test results, actively pursue testing on a commercial scale.

Coordinate and refine petrographic and chemical characterization in support of the AF development effort.

- * Develop and test a comprehensive petrographic "liberation analysis" approach to evaluate all major minerals other than pyrite.
- * Carry out extensive chemical and mineralogical analyses to accurately ascertain the effectiveness of AF.
- * Conduct appropriate grinding studies and establish optimum liberation criteria.

INTRODUCTION

The Illinois coal industry is struggling to compete in local and world markets with out-of-state coal that is lower in sulfur. Competition with western USA coal producers is intense and will continue to increase with implementation of pending legislation requiring lower SO₂ emissions. Since the great majority of Illinois Basin coals range from 2% to 5% total sulfur, and on average are roughly equal in the distribution of inorganic (pyritic) and organic sulfur contents, physical and chemical methods which remove both forms must often be used to achieve compliance coals. Current accepted practice is to remove SO₂ produced during combustion by means of flue gas desulfurization which is capital intensive and costly in operation.

Those methods involving the removal of the organic form of sulfur are generally expensive, since they involve use of chemicals, heat, pressure or otherwise severe conditions. Commercial methods used for the limited removal of coarser pyritic sulfur are fairly cost effective but can be relatively inefficient. For instance, gravity based methods often remove coarse pyrite (+6 mesh) but leave the finely disseminated mineral matter entrapped within the coarse clean coal fraction. Even the present methods available for fine sized coal that are based on surface properties tend to be limited with regard to either cost or the efficiency of separation.

While the processing by froth flotation of 28x0 mesh feed in domestic coal preparation plants has become more prevalent and accepted with increased

mining mechanization, it has been employed primarily for fines recovery (28x100 mesh) and for reducing the ash forming minerals in the fines. Recovery of this fraction has become both an economical and environmentally sound procedure for recovery of marketable product representing up to 20% of the plant feed. Unfortunately, traditional flotation of this size feed does not significantly reduce the pyritic sulfur in Illinois coals.

Unlocking enclosed pyrite and ash from coal by extensive comminution to ultrafine sizes (<400 mesh), will yield a product which can be difficult to separate from mineral matter on either a wet or dry basis. Numerous wet processes for separation of ultrafine coal from mineral matter include oil agglomeration (Capes et al., 1982; Constantin et al., 1980; Theodore, 1985), selective flocculation (Attia et al., 1983; Attia et al., 1985), coal-pyrite flotation (Miller, K., 1973), and dry processes such as high gradient magnetic separation (Mathieu, 1982; Kester, 1966; Liu, 1978). All have been reported to be successful at reducing pyritic sulfur in coal. Nevertheless, due to the need for extensive retrofits which have not been tested at full scale, it is felt by some in coal preparation research that some variation of the froth flotation process, which is currently a viable and widely accepted method, will ultimately serve as a model for removal of pyrite and ash from ultrafine coal. Current obstacles which prevent its immediate implementation are discussed in more detail and to provide the reader with the inherent limitations as they currently exist.

A significant problem in floating ultrafine coal is the decreased flotation rate and resulting decrease in recovery (Yoon, 1984; Zimmerman, 1979). In a flotation process, the coal particle must first collide with a rising air bubble and second, surface interactions must be such that the particle adheres to the air bubble as it rises to the collection zone (Fuerstenau, 1980). The probability that a particle and bubble will collide is adversely affected by smaller size, lower mass to size ratio (Flint and Woarth, 1971), surface charges on the particles and streamlines around the bubble (Fuerstenau, 1980). A decrease in recovery at fine sizes is due in part to a reduced probability of bubble/particle collision. Additionally, reagent selectivity, which is based on hydrophobic/hydrophilic interactions between the flotation reagent and the coal surface in an aqueous environment, can significantly influence the overall effectiveness of the coal flotation process. Adhesion of particles to bubbles is not only affected by these interactions but by the natural hydrophobicity of the coal particle, induction time, balance between bubble growth and coalescence, and surface tension. The flotation of ultrafine coal can therefore be significantly influenced by many parameters.

Improved control of the rate of flotation by employing microbubbles has been reported for ultrafine size coal (Yoon, 1984). Introduction of externally generated microbubbles into a column of ultrafine coal gave improved recovery and product quality as compared to conventional sub-aeration cells. However, considering the widely divergent frother dosages which were used for the comparison, the actual effectiveness of the results is debatable.

The concept of microbubble flotation, nonetheless, does attempt to take advantage of the differences in particle and bubble diameter dependencies (Jameson et. al, 1977). Yet as coal particles become smaller, not only

will the specific surface area increase significantly, but surface charges and chemical interactions with the reagent become important. Microbubbles, in and of themselves, may not completely control effective flotation of ultrafine coal. Thus, the ISGS has developed and tested a physical method (based on principles both from froth flotation and oil agglomeration) which has the potential to become more efficient as the particle size of the coal is reduced. This approach, now called the ISGS Aggregate Flotation Process, incorporates chemical control of bubble size with chemical control of particle interactions in the flotation of ultrafine coal. Rather than produce bubbles external to the cell, they are generated "in-situ" and their size is controlled by specific surfactants. Additionally, the surfactant is used in conjunction with an alcohol or oily collector such as kerosene. This aids in increasing both the specific surface area of the alcohol reagent and kerosene by emulsification and enhances the bond attachment of the apolar agent. With enhanced stability of collector attachment, the adhesive bond (chemisorptive) of the coal particles to the air bubbles is strengthened resulting in improved recoveries. With the preferential adsorption onto the coal surfaces, both ash and pyrite rejection are subsequently improved.

Results to date indicate substantial potential exists for removing the highly disseminated pyrite in many coals by grinding it until apparent optimum pyrite liberation occurs, as indicated from petrography, and then selectively floating coal with foaming surfactants which promote the stabilization of microbubbles and the formation of coal-air aggregates. For most of the coals tested very good Btu recoveries (80%-90%) with excellent rejections of pyrite and ash (70%-90%) have been obtained utilizing equipment presently available in conventional froth flotation. The initial economic evaluation appears encouraging.

Thus the major goal of the project has been the development and evaluation of a new and evolving ultrafine coal cleaning process - ISGS Aggregate Flotation (AF). A very important second goal has been the development and utilization of a variety of petrographic, mineralogic and chemical methods for the thorough characterization of products from the cleaning processes. By integrating these two goals progress has been made in understanding and improving the process.

EXPERIMENTAL

Reagent selection in the Aggregate Flotation process has evolved from bench scale tests utilizing the old AF method (premixed foam, column settling, whole ROM coals). Several surfactants yielded improved results regarding ash and pyrite rejection and increased Btu recovery. Upon batch testing in traditional (sub-aeration) flotation units, these "preferred" commercial surfactant/alcohol mixtures were shown to be superior with respect to lower dosages required for equivalent Btu recovery when compared to conventional alcohol based frothers. The major disadvantage of such commercially prepared proprietary mixtures was that its composition was unknown and formulated for other end-use applications (oil-drilling). While these reagents were shown to produce improved results with respect to Btu recovery, the specific composition was not optimum for rejection of ash and pyrite in ultrafine coal. To accurately compare pure reagents of known

composition for effectiveness and those for surfactants/collector mixtures, a standardized AF (batch) flotation test was designed, and is detailed in the AF Flotation Procedure. A comprehensive description of feed coal samples used throughout this study is given in Appendix A.

AF Flotation Procedure

The AF tests include the use of techniques such as rod/ball or stirred-ball milling for preparation of feed coal of various size distributions, such as -28 mesh for fine coal or 80% -400 mesh for ultrafine coal. A suitable pulp density ranging from 3.5 wt.% to 4.5 wt.% solids is prepared and a measured volume of slurry is placed into a 4 liter, Denver D-2 sub-aeration cell, and the impeller speed set to 1075 rpm. For thorough mixing the slurry is agitated two minutes. After this time a known quantity of reagent (lbs active/ton dry solids basis) is added and mixed for 2 minutes. At the end of 2 minutes, air is introduced into the cell at a rate of 8 scfh. A coal-laden froth is produced and collected by scraping it by hand from the top of the cell into collection receivers. Collection continues for a total of 8 minutes with fractions being collected at given time intervals. The solids and liquids from the froth concentrates and tailings are separated by filtration, dried, weighed, and analyzed for moisture, ash, total sulfur (TS), forms of sulfur including pyritic, sulfatic and organic (OS, by difference), and heating value (Btu/lb). Percent weight (yield), Btu recovery, and ash and pyrite rejection values are then calculated.

Experimental Approach Used to Attain Objectives

Investigations for pure commercial foaming surfactants, as a function of dosage versus Btu recovery and rejection of ash and pyrite, were undertaken during the contract year for a number of coal samples. This study included efficiency as a function of structure (i.e., straight and branched chain sulfated ethers, linear alpha olefins, alkyl/aryl sulfonates, ethoxylates, etc.) type (anionic, non-ionic, cationic) and cost effectiveness. Similar tests were also conducted for prepared mixtures of known surfactants with frothing alcohols. Attempts were also undertaken to develop proprietary reagent mixtures. Such an approach was considered after examination of the pure surfactants, with a statistical optimization method being employed for such formulations.

The most widely used collectors employed in conventional coal flotation processes are fuel oil and kerosene which are often used in conjunction with alcohol based frothers. The AF process may also utilize kerosene, but with a surfactant. Tests were initiated with other non-oily collectors to compare the efficiency of alternate compounds. Such collectors included tall and tar oils and fatty acid mixtures, all considered to be by-products with potential for cost effectiveness. A statistical optimization method for surfactant/collector dosage interactions was initiated, but not completed. The substitution of corn and soybean oils (vegetable) for mineral oils was also included in this study with preliminary tests indicating they are quite competitive in both performance and cost.

As the practice of Aggregate Flotation gains momentum as a means to remove mineral matter and pyrite from Illinois coals, appropriate equipment

selection may be critical. While the AF process has shown that microbubbles are essential for fine and ultrafine coal flotation, it has been predominantly batch tested on Denver sub-aeration cells. To differentiate advantages in cell design, tests were conducted on alternate "microbubble" units. Comparative tests for results obtained by AF versus those from column flotation were arranged through the CRSC via the United Coal Company (Bristol, VA). For this test the Illinois No. 5 (Springfield-SE) sample was used. ISGS compared batch results obtained in a conventional Denver cell to those obtained in a Wilfley-Weber microbubble unit. For the latter test, all process conditions were kept equal, as well as the type of surfactant employed. Again the Illinois No. 5 (Springfield-SE) sample was used.

The continuous flow unit (CFU, 10-30 lb/hr) was designed to achieve several goals which cannot be achieved by batch flotation testing. First and foremost was confirmation, on a continuous flow basis, of results obtained in batch testing with respect to optimized particle size, reagent type, aeration rate and retention times. It was anticipated that a number of CFU runs would be conducted this year; however, due to basic equipment needs for successful operation of the unit, efforts in this area were delayed and will be undertaken in the new contract year.

Options for final product form and costs associated with such processes as coal-water slurry (CWS), briquetting or pelletization were assessed. A brief literature review is presented which discusses AF final product options.

Pilot plant testing of the AF process is also being actively pursued. After considerable effort, a DOE proposal was submitted for design, construction and testing on the one TPH level at the Homer City (PA) Clean Coal Test Facility (CCTF). The proposal was, however, rejected. Efforts are currently underway to test the ISGS reagent package in the Bechtel Microbubble process at the CCTF. Depending upon positive results, testing at a full scale level is anticipated, perhaps thru a consortium which includes, ISGS, DENR, and a major Illinois Coal producer.

RESULTS AND DISCUSSION FOR BASIC AND APPLIED AF STUDIES

Comparison of Surfactant Efficiency Based on Structure, Type and Cost-Effectiveness

The Aggregate Flotation (AF) process, an integrated variant of the conventional oil agglomeration and froth flotation technologies, has been demonstrated to have significant advantages over either technology alone for removing pyritic sulfur and ash from difficult-to-clean Illinois Basin coals. The production of microbubbles having unique collection characteristics especially tailored for removing pyrite and ash from ultrafine coal (80% -400 mesh) is an important feature of the AF process, since proprietary reagents are used to generate these microbubbles. The AF process works in typical sub-aeration cells, and does not require new cell designs for producing the microbubbles. Results using a number of these proprietary reagents on three Illinois Basin Coals (No. 5, No. 2, No. 6) are given in Tables 1 to 6. (Note that portions of the data presented are in terms of the flotation product, which in some instances is derived from convention-

ally prewashed feeds.) The standard conditions as described in the Experimental section were employed for each test. By holding several variables constant (% solids, impeller speed, aeration rate, conditioning time) and performing single stage dosage/recovery tests, different reagents could be compared and accurately evaluated for cost/benefit effectiveness. All reagents tested were assigned laboratory numbers to protect the confidentiality of the manufacturer, if commercial, and chemical composition if developed at ISGS or Southern Illinois University.

From the comprehensive data obtained in this study for the washed Illinois No. 5 coal (Springfield-SE) it can be seen from Tables 1 and 2 that an additional 45% to 50% rejection of ash and pyrite at >80% Btu recovery can be attained if the washed product is treated by the AF process. Similar work as shown in Table 3 has also been conducted for the difficult-to-float, oxidized, Illinois No. 2 (Colchester-W) ROM coal. In this instance, extrapolated ash and pyrite rejection on the order of 60% at the 80% Btu level is expected. For the ROM Illinois No. 5 (Springfield-SE) coal, further evidence for high pyritic sulfur and ash rejection is given in Table 4, where 70% to 80% ash and pyrite rejection at 90% Btu recovery is shown. Results for the Illinois No. 6 (Herrin-SW) coal given in Table 5 and 6 are similar.

While it is somewhat difficult to show substantial improvement in reduced reagent consumption for the Illinois No. 5 (Springfield-SE) washed coal, there is evidence that several surfactants employed are more effective than MIBC. These include for example, 1008-11-26, -31, -44, -45, -50, and 1008-12-1, which if extrapolated to equivalent MIBC dosages, yield higher Btu recovery (ash and pyrite rejection would be higher due to the higher Btu recovery). This is particularly noticeable for the Illinois No. 2 (Colchester-W) ROM coal where MIBC dosages are cost prohibitive, but several of the proprietary reagents have potential for cost-effectiveness.

Comparison of Surfactant and Surfactant/Collector Efficiency Based on Structure and Type

In a concerted effort to find better reagents for use in the Aggregate Flotation Fine Coal Cleaning process, specific compounds synthesized by Dr. C. Meyers of the SIU Chemistry Department have been systematically tested and evaluated for possible utilization. Structure/property correlations have been obtained for commercially available compounds and for a class of newly synthesized ones, regarding their effectiveness for flotation of ultrafine coal.

As shown in Table 7 and 8 for the Illinois No. 5 (Springfield-SE) coal (hva), those commercial reagents tested indicated that chain length, total carbon and molecular structure were most influential regarding dosage/recovery behavior. One of the synthesized reagents was shown to be the most effective when compared to all other commercial ones or frothing alcohols such as MIBC. Significant reductions in dosage to yield equivalent Btu recovery were observed with this new reagent. In some instances reduction in dosage by a factor of 2 or more were realized.

Although the synthesized reagents were not as ash and pyritic sulfur re-
jective as the alcohols tested, mixtures of these compounds with such alcohols produced a synergistic effect (Table 9). Both lower dosages of each

Table 1 Reagent Study for Illinois No. 5 (Springfield-SF) washed coal

Reagent	Dosage ¹	Recovery,		Ash, %		Pyritic sulfur, %		Rejection, %	
	#A/tds	wt	BTU	Feed	Product	Feed	Product	Ash	Pyritic S
1008-11-									
-1	1.4	28.3	31.3	11.9	3.7	1.11	0.36	91.3	91.3
	1.6	70.9	77.0	11.3	4.6	1.14	0.65	72.9	60.7
	1.9	75.3	81.8	11.9	5.0	1.08	0.71	68.4	50.5
	2.1	88.8	95.0	11.7	6.2	1.17	0.82	52.9	37.7
-9	1.6	73.7	79.4	12.8	6.2	1.06	0.74	65.6	53.3
	1.8	89.4	94.0	11.4	5.6	1.16	0.77	45.8	35.3
-26	1.4	90.2	96.9	11.4	6.3	1.13	0.81	57.7	42.7
	1.8	92.4	98.4	11.1	5.9	1.06	0.75	50.9	34.6
-29	1.8	47.8	52.3	11.1	3.7	1.06	0.48	84.1	78.5
		89.0	96.0	11.2	5.2	1.07	0.72	58.5	42.4
H18C	1.5	64.7	67.0	11.5	4.2	1.19	0.60	77.5	68.7
	1.6	77.0	83.8	11.3	4.6	1.13	0.57	70.5	62.5
	2.5	87.4	94.6	11.4	4.9	1.14	0.66	62.4	49.1
-32	1.8	72.0	77.8	11.3	4.7	1.09	0.69	70.1	59.0
	2.4	89.1	95.4	11.0	5.2	1.00	0.67	57.8	38.5
-39	1.4	74.5	79.5	11.2	5.5	1.08	0.67	63.4	53.8
	1.6	79.5	85.1	11.6	6.0	1.23	0.81	58.8	47.6
	1.8	85.9	91.2	11.2	6.0	1.12	0.77	54.0	40.9
-44	1.2	60.1	65.1	11.9	4.8	1.20	0.69	76.2	65.8
	1.4	90.1	93.9	11.4	5.9	1.16	0.78	58.3	51.6
-45	1.0	80.5	86.0	11.1	5.5	1.04	0.65	60.1	49.7
	1.2	86.5	92.4	11.1	5.6	1.08	0.72	56.4	38.7
	1.4	92.9	99.4	11.0	6.6	1.02	0.74	44.3	32.6

¹ #A/tds lbs active reagent/ton dry solids

As mined coal: 27.1% ash, 2.37% pyritic sulfur, 10450 BTU

Table 2 Reagent Study for Illinois No. 5 (Springfield-SF) Washed Coal

Reagent	Dosage ¹	Recovery,		Ash, %		Pyritic sulfur, %		Rejection, %	
	#A/tds	wt	BTU	Feed	Product	Feed	Product	Ash	Pyritic S
1008-11-50	0.8	81.5	85.2	9.6	5.5	1.18	0.92	53.3	36.1
	1.0	88.1	92.2	9.5	5.7	1.08	0.83	47.3	32.4
1008-12-1	1.2	64.0	69.6	11.5	4.2	1.14	0.56	77.3	68.5
	1.4	68.9	74.3	11.2	4.6	1.02	0.56	71.6	62.2
	1.6	90.1	96.8	11.1	5.1	1.05	0.65	58.7	43.9
1008-11-39	1.4	74.5	79.5	11.2	5.5	1.08	0.67	63.4	53.8
	1.6	79.5	85.1	11.6	6.0	1.23	0.81	58.8	47.6
	1.8	85.9	91.2	11.2	6.0	1.12	0.77	54.0	40.9
-47	2.5	64.7	69.4	11.5	5.3	1.18	0.73	70.2	70.8
-49	3.5	90.4	93.8	9.7	6.5	0.99	0.80	37.1	24.4
-48	2.5	62.2	66.8	12.1	5.9	1.11	0.70	69.8	60.8
	3.3	80.7	84.2	9.7	6.2	1.01	0.82	48.1	34.6
-16	3.3	76.8	82.2	11.4	5.8	1.17	0.77	58.9	48.3
-40	2.6	32.6	35.7	12.1	4.5	1.13	0.54	87.8	84.7
	5.0	83.4	88.4	11.6	6.8	1.16	0.85	51.1	38.8
-43	4.7	61.9	66.5	11.3	5.4	1.12	0.73	70.4	59.7
-42	5.0	55.5	60.2	12.2	5.4	1.13	0.66	75.0	67.6
-41	5.0	30.0	32.6	11.1	4.1	1.15	0.51	88.9	87.0

¹ #A/tds - lbs active reagent/ton dry solids

As mined coal: 27.1% ash, 2.37% pyritic sulfur, 10450 BTU

Change in Feed Ash (-11.5 to 9.7) is due to new split of washed sample

Table 3 Reagent Study for Illinois No. 2 (Colchester-W) ROM Coal

Reagent	Dosage ¹	Recovery, %		Ash, %		Pyritic sulfur, %		Rejection, %	
	#A/tds	wt	BTU	Feed	Product	Feed	Product	Ash	Pyritic S
1008-11-									
-50	4.1	86.6	91.0	10.5	5.8	3.26	1.90	52.6	50.1
	5.6	92.9	96.7	10.5	7.1	3.22	2.16	37.4	37.6
-39	6.4	19.2	20.8	10.7	4.4	3.30	1.06	92.1	93.9
	7.7	85.8	90.5	10.7	6.7	3.34	2.06	46.1	47.0
	8.0	87.7	92.6	10.2	6.0	3.23	1.98	48.4	46.0
-31	7.5	69.3	75.9	10.5	3.5	3.32	1.02	76.9	78.7
	8.5	87.1	94.4	10.5	4.5	3.32	1.40	62.6	63.4
MIBC	11.3	68.9	76.4	10.6	3.8	3.80	0.98	75.0	81.9
-45	4.8	85.4	92.3	10.5	4.4	3.17	1.25	63.8	66.3
	5.1	89.2	95.6	11.1	5.7	2.98	1.36	54.7	59.1
-48	8.9	86.2	86.1	11.2	6.65	3.04	1.78	51.9	52.5
	13.9	87.2	91.8	10.8	7.0	3.50	2.18	43.6	45.8
-49	10.7	82.9	86.1	11.2	8.19	2.99	2.06	39.3	43.1

¹#A/tds - lbs active reagent/ton dry solids

Table 4 Reagent Study for Illinois No. 5 (Springfield-SE) ROM coal

Reagent	Dosage ¹	Recovery, %		Ash, %		Pyritic sulfur, %		Rejection, %	
	#A/tds	wt	BTU	Feed	Product	Feed	Product	Ash	Pyritic S
1008-11-									
-31	1.6	57.27	78.6	29.3	5.6	2.71	0.87	88.9	81.7
	1.7	66.09	89.0	28.4	6.3	2.57	0.92	85.3	76.4
	2.0	71.4	96.4	30.1	8.6	2.67	1.16	79.6	69.0
-26	1.55	62.15	85.4	28.9	5.9	2.55	0.79	87.3	80.8
	2.0	73.9	99.0	30.2	9.8	2.73	1.39	76.0	62.3
-45	1.0	45.9	58.5	28.8	7.0	2.43	0.71	88.4	85.6
	1.25	56.4	75.0	28.8	7.3	2.27	0.75	84.0	78.6
	1.5	70.7	94.4	30.9	10.7	2.67	1.46	75.5	61.5
-52	1.30	70.2	95.6	30.8	10.1	2.56	1.35	76.8	62.3
-53	1.25	70.4	94.9	30.8	9.8	2.49	1.35	77.7	61.8
-54	1.25	69.5	92.9	30.1	9.5	2.52	1.26	78.1	65.1
-56	1.4	68.9	92.9	29.7	7.9	2.41	1.03	81.5	70.4

¹#A/tds - lbs active reagent/tds

Table 5 Reagent Study for Illinois No. 6 (Herrin-SW)* Washed Coal

Reagent	Dosage ¹ #A/tds	Recovery, %		Ash, %		Pyritic sulfur, %		Rejection, %	
		wt	Btu	Feed	Product	Feed	Product	Ash	Pyritic S
1008-11-50	2.1	78.6	87.9	17.8	8.9	1.37	1.10	60.6	36.9
1008-11-26	4.8	54.9	63.9	17.6	5.1	1.35	0.69	84.0	72.0
	9.1	91.9	93.4	17.7	7.1	1.37	0.97	67.3	42.2
1008-11-31	2.7	25.8	30.0	18.8	5.7	1.41	0.56	92.2	89.7
	6.4	75.5	86.4	17.7	6.8	1.32	0.83	70.9	52.7
1008-11-44	3.0	71.3	80.0	17.0	7.5	1.48	1.02	68.6	51.0
	6.3	92.3	98.5	17.5	12.4	1.51	1.39	34.6	15.0
	7.1	88.7	95.3	17.6	11.9	1.25	1.13	39.9	19.7
1008-11-56	6.3	77.7	88.4	17.7	7.2	1.24	0.84	68.5	47.5

1 lbs active/ton dry solids

Table 6 Reagent Study For Illinois No. 6 (Herrin-SW)* ROM Coal

Reagent	Dosage ¹ #A/tds	Recovery, %		Ash, %		Pyritic sulfur, %		Rejection, %	
		wt	Btu	Feed	Product	Feed	Product	Ash	Pyritic S
1008-11-44	3.7	68.7	91.4	34.9	14.9	2.28	1.65	70.6	50.1
1008-11-56	5.9	54.2	80.4	38.4	11.1	2.58	1.85	84.4	61.2
1008-11-26	7.4	68.9	89.0	37.8	10.2	2.37	1.22	84.1	69.6
1008-11-31	7.5	60.8	77.5	38.4	9.1	2.48	1.11	88.0	77.2

*IBCSP No. 4; lbs active/ton dry solids

Table 7. Commercial Surfactant Composition, Dosage versus Recovery, and Resulting Ash and Pyritic Sulfur Rejection Results for Springfield No. 5 Coal

Notebook No.	General Chemical Formula	Avg. MW	Dosage #A/Tds m/Tds	Molar Ratio	% Recovery Wt. %	% Ash Feed	% PS Feed	% PS Product	% Ash Product	% Rejection Ash	% Rejection Pyrite
1008-11-40	Commercial	420	2.6 5.0	1.19 2.15	32.6 83.4	35.7 88.4	12.1 11.6	1.08 1.16	4.5 6.8	0.58 0.85	84.7 38.8
1008-11-43	Commercial	600	4.0	3.55	61.9	66.5	11.1	1.14	5.2	0.74	59.7
1008-11-42	Commercial	690	5.2	3.29	55.5	60.2	12.2	1.14	5.4	0.68	67.6
1008-11-41	Commercial	950	5.2	2.39	30.0	32.6	11.1	1.05	4.1	0.51	87.0
1008-11-39	Commercial	288.4	1.4 1.6 1.8	2.20 2.51 2.83	74.5 79.5 85.9	79.5 85.1 91.2	11.2 11.7 11.2	1.08 1.10 1.12	5.5 6.0 6.0	0.67 0.65 0.77	53.8 47.6 40.9
1008-11-47	Commercial	272	2.5	4.17	64.7	69.4	11.4	1.17	5.8	0.77	70.8
1008-11-16	Commercial	420	3.3	3.56	76.8	82.2	11.4	1.15	5.8	0.80	48.3

*Molar Ratio - Moles of surfactant relative to moles of standard, 1008-11-39, at 1.6 lb/ton dry solids

#A/Tds = lbs active surfactant/ton dry solids

m/Tds = moles surfactant/ton dry solids

All results on a dry basis.

Table 8. Dosage/Recovery and Analytical Results for Springfield No. 5 Coal by the Aggregate Flotation Process.

NOTEBOOK NO.	Reagent	Dosage #A/tds	M/tds	Molar Ratio*	% Recovery Wt	% Recovery BTU	% Ash Feed	% Ash Product	% PS Feed	% PS Product	AR	PR
1008-11-50	Synthetic	0.8	1.25	0.50	81.5	85.2	9.6	5.5	1.18	0.92	53.3	36.1
		1.0	1.58	0.63	88.1	92.2	9.5	5.7	1.08	0.83	47.3	32.4
1008-11-51	Synthetic	1.0	1.97	0.78	75.1	80.3	10.9	5.3	1.04	0.67	63.2	51.2
		1.2	2.37	0.94	80.3	85.0	10.2	5.6	1.01	0.70	55.6	44.7
1008-12-1	HR100 Alcohol	1.2	4.19	1.67	64.0	69.6	11.5	4.2	1.14	0.56	77.3	68.5
		1.4	4.89	1.94	68.9	74.3	11.2	4.6	1.02	0.56	71.6	62.2
		1.6	5.59	2.23	90.1	96.8	11.1	5.1	1.05	0.65	58.7	43.9
1008-11-39	Commercial	1.4	2.20	0.89	74.5	79.5	11.2	5.5	1.08	0.67	63.4	53.8
		1.6	2.51	1.00	79.5	85.1	11.6	6.0	1.23	0.81	58.8	47.6
		1.8	2.83	1.13	85.9	91.2	11.2	6.0	1.12	0.77	54.0	40.9
1008-12-2	MIBC Alcohol	2.1	9.34	3.72	66.8	72.5	12.4	3.8	1.28	0.58	79.5	69.7
		2.4	10.68	4.25	87.4	94.7	11.4	4.9	1.14	0.66	62.4	49.1
1008-11-49	Synthetic	3.2	4.43	1.76	85.4	88.5	9.8	6.7	0.98	0.79	42.0	31.1
		3.5	4.84	1.93	90.4	93.8	9.7	6.5	0.99	0.80	37.1	24.4
1008-11-48	Synthetic	2.5	3.31	1.32	62.2	66.8	12.1	5.9	1.11	0.70	69.8	60.8
		3.3	4.38	1.75	80.7	84.2	9.7	6.2	1.01	0.82	48.1	34.6

#A/tds = lbs active reagent/ton dry solids; M/tds = moles reagent/ton dry solids

AR = Ash Rejection; PR = Pyrite Rejection

HR100 is a proprietary alcohol reagent from Union Carbide Corporation

Note: Changes in feed ash were due to a new split of Springfield tabled coal

* Molar Ratio - moles of reagent/ton dry solids relative to moles of standard, 1008-11-39 at 1.6 lb/ton dry solids

Table 9 Dosage, Recovery, and Grade for Mixtures in Flotation of Ultrafine-coal for the Springfield-SE (Ill. No. 5) Sample.

Reagent	Dosage # A/tds	% Wt Rec	% BTU Rec	% Ash Feed	% Ash Product	% PS Feed	% PS Product	% Ash Rej	% Py Rej
1008-11-50	0.8	81.5	85.2	9.6	5.5	1.18	0.92	53.3	36.1
	1.0	88.1	92.2	9.5	5.7	1.08	0.83	47.3	32.4
HR100	1.2	64.0	69.6	11.5	4.2	1.14	0.53	77.3	68.5
	1.4	68.9	74.3	11.2	4.6	1.02	0.56	71.6	62.2
	1.6	90.1	96.8	11.1	5.1	1.05	0.65	58.7	43.9
0.6 #A/tds : 1008-11-50 + 0.6 #A/tds : HR100	1.2	85.5	91.7	10.1	5.2	1.14	0.76	55.5	42.1
0.3 #A/tds : 1008-11-50 + 0.9 #A/tds : HR100	1.2	73.1	78.1	10.0	4.3	1.03	0.64	68.4	55.8

#A/tds - lbs active reagent/ton dry solids

component in the mixture, as compared to either single component alone, produced higher Btu recovery with increased ash and pyritic sulfur rejection.

For the Illinois No. 2 (Colchester-W) coal (hvc), the synthesized compounds again proved to be the most effective (Table 10). Significant reductions in dosage to yield equivalent Btu recovery were also observed with this reagent. In this instance reduction by a factor of 3 or more was realized when compared to the commercial compounds. When compared to the alcohol frother MIBC, the synthesized reagent was on the order of 7 times more effective. Hence, optimum structure/performance for this new class of reagents may be directly related to the rank of the coal. This aspect, however, requires further evaluation.

More recent work at ISGS on an Eastern coal (Upper Freeport, ROM, 80% -65 mesh) has shown benefits for such new reagents in conventional froth flotation processes. (Froth Flotation cell conditions employed are more rigorous than those in Aggregate Flotation. Particle size differences for the two processes are also different, i.e. 28x0 vs -400 mesh. The reagent for flotation of 28x0 feed, generally for Eastern coals, is MIBC, whereas AF uses proprietary surfactant/alcohol/kerosene blends.)

Using a statistically designed test for combinations of MIBC and 1008-11-50 (Table 11) yielded typical surface response diagrams as shown in Figures 1 to 5. For example, from Figure 1 at a total dose of 0.4 lbs MIBC/tds alone, a Btu recovery of ~ 79% is projected. However, at a total dosage of 0.4 lbs/tds which consists of 0.2 lbs 1008-11-50 + 0.2 lbs MIBC, the Btu recovery is increased to ~95%. Similarly, one can predict the resulting ash and pyritic sulfur rejection values from Figures 2 and 3. Additional supporting evidence is given in Figures 4 and 5 where Btu recovery, ash and pyrite rejection are plotted as a function of a fixed total dosage (0.25) or fixed fraction of 1008-11-50. Again, indications are that higher Btu recovery, with good ash and pyritic sulfur rejections are obtained with mixtures of these surfactants with MIBC for conventional size flotation feed (28x0).

Additional efforts in optimizing structure/rank dependencies, investigations into the synergism of mixtures and other synthetic derivatives, are being undertaken in the next contract period.

Kinetic Effects By Specific Surfactants

In conjunction with the reagent study, a series of batch kinetic tests (recovery/grade vs time) were performed for a number of different surfactants. The goals were the definition of acceptable cell retention times which could then be applied to the continuous flow unit and an investigation of the potential for a new concept, "surfactant controlled rate of flotation" and its potential validity for currently existing plant circuitry. In order to accurately ascertain these aspects, the standard ISGS flotation test was employed. All data summarized here were obtained at a fixed dosage of 1.6 lbs active/ton dry solids, with the Illinois No. 5 (Springfield-SE) washed coal, stirred ball mill ground to 80% -400 mesh, serving as the initial test sample.

Table 10 Dosage/Recovery and Analytical Results
Colchester No. 2 Coal by the Aggregate Flotation Process.

Notebook No.	Reagent	Dosage		Molar Ratio*	% Recovery		% Ash		% PS		% Rejection	
		#A/tds	M/tds		Wt	BTU	Feed	Product	Feed	Product	AR	PR
1008-11-51	Synthetic	2.4	4.73	0.39	78.6	84.0	11.6	6.1	4.81	2.78	58.9	54.6
		3.1	6.12	0.51	86.7	92.5	10.5	6.3	4.83	3.01	52.3	45.8
1008-11-50	Synthetic	4.1	6.51	0.54	86.6	91.0	10.5	5.8	3.26	1.90	52.6	50.1
		5.5	8.73	0.72	92.9	96.7	10.5	7.1	3.22	2.16	37.4	37.6
1008-12-1	HR100 Alcohol	7.5	26.2	2.17	69.3	75.9	10.5	3.5	3.32	1.02	76.9	78.7
		8.5	29.7	2.45	87.1	94.4	10.5	4.5	3.32	1.40	62.6	63.4
1008-11-39	Commercial	6.6	10.4	0.86	19.2	20.8	10.7	4.4	3.30	1.06	92.1	93.9
		7.7	12.1	1.00	85.8	90.5	10.7	6.7	3.34	2.06	46.1	47.0
		8.0	12.6	1.04	87.7	92.6	10.7	6.0	3.23	1.98	48.4	46.0
1008-12-2	MIBC Alcohol	10.8	48.1	3.97	68.9	76.2	10.6	3.8	3.79	0.98	75.3	82.1
		14.3	63.5	5.25	79.2	85.0	10.5	4.9	2.89	1.19	62.7	67.4
1008-11-49	Synthetic	8.8	12.2	1.01	63.8	70.9	11.1	5.5	3.36	1.73	67.5	66.1
		13.0	18.0	1.49	87.5	91.8	10.7	6.9	3.50	2.18	43.6	45.6
1008-11-48	Synthetic	14.1	18.7	1.54	82.1	87.9	11.0	9.4	3.26	2.79	26.8	26.5

#A/tds = lbs active reagent/ton dry solids; M/tds = moles reagent/ton dry solids

AR = Ash Rejection; PR = Pyrite Rejection

HR100 is a proprietary alcohol reagent from Union Carbide Corporation

Note: Changes in feed ash were due to a new split of Colchester tabled coal

* Molar Ratio - moles of reagent/ton dry solids relative to moles of standard, 1008-11-39 at 7.7 lbs active/solids

Table 11 RESULTS FOR UPPER FREEPORT (ROM 28X0) USING MIBC/SIV SYNTHETIC REAGENTS

Reagent	Dosage ¹ #A/tds	Recovery, %		Ash, %		Pyritic sulfur, %		Rejection, %	
		Wt	BTU	Feed	Product	Feed	ProductAsh	Pyritic S	
1008-11-30*	0.20	51.1	65.2	23.9	5.5	0.99	0.23	88.2	88.1
	0.40	60.9	76.1	24.0	7.1	0.97	0.31	82.0	80.6
1008-11-30*	0.10	53.1	66.8	23.5	5.7	0.92	0.21	87.1	87.9
1008-11-50	0.10								
1008-11-30	0.10	75.4	89.6	22.9	9.9	0.88	0.47	67.4	59.4
1008-11-50	0.20								
1008-11-30*	0.20	63.9	78.8	23.9	7.9	0.94	0.35	78.8	76.2
1008-11-50	0.10								
1008-11-30*	0.20	61.1	76.4	23.8	6.9	0.96	0.31	82.2	80.2
1008-11-50	0.10								
1008-11-31*	0.10	72.2	87.8	24.1	9.8	0.96	0.32	70.5	75.9
	0.15	80.9	95.6	23.9	11.7	0.93	0.40	60.2	65.4
1008-11-50*	0.20	62.2	74.0	20.9	7.5	0.84	0.36	77.6	73.5
	0.19	62.4	78.7	25.2	7.9	1.00	0.37	80.5	76.9
	0.29	75.1	89.5	23.2	10.2	0.94	0.52	67.0	58.3
DOW-150	0.30	71.8	87.2	23.6	9.2	0.96	0.49	72.1	63.5
NALCO 8834*	0.30	57.2	70.9	23.5	7.3	0.95	0.32	82.2	80.7

* The product is compliance coal for plants built 1971-1978

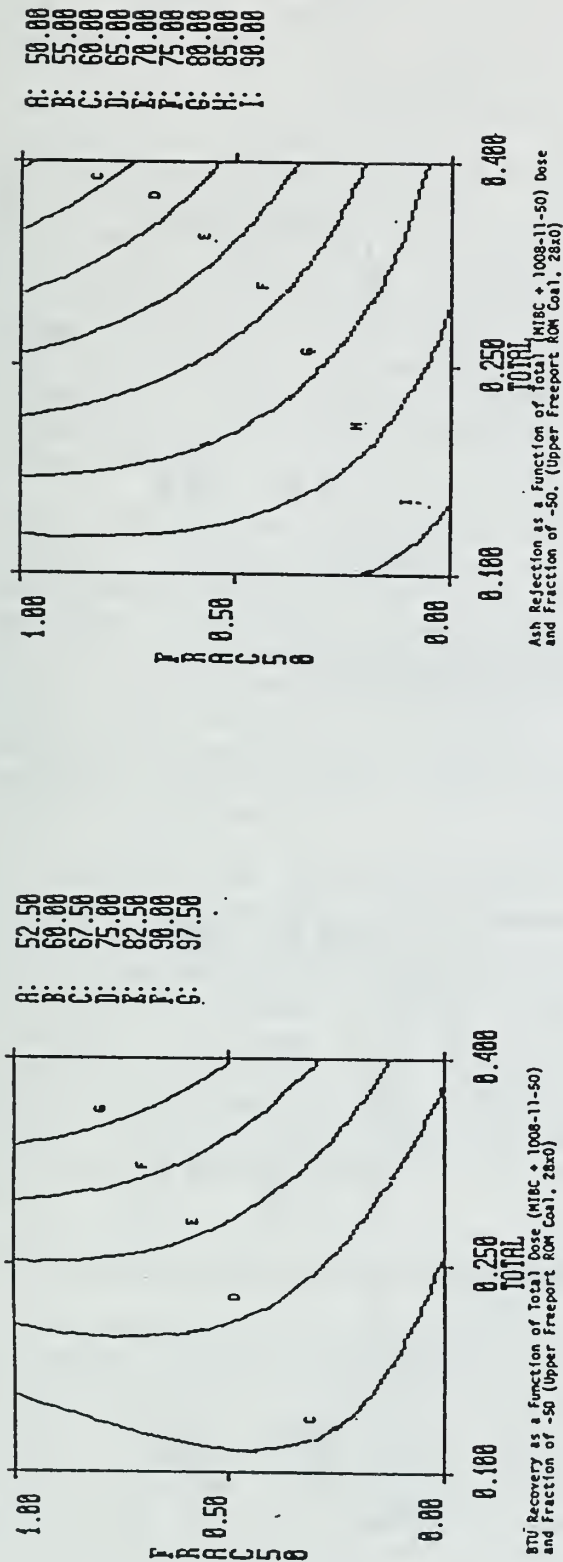


Figure 1

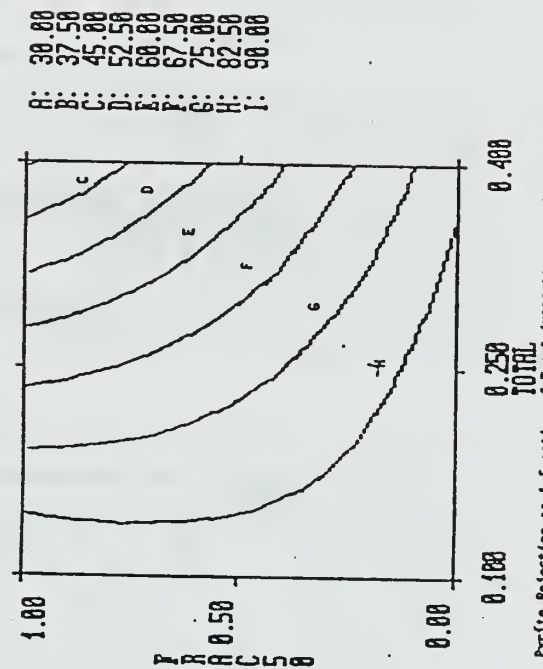
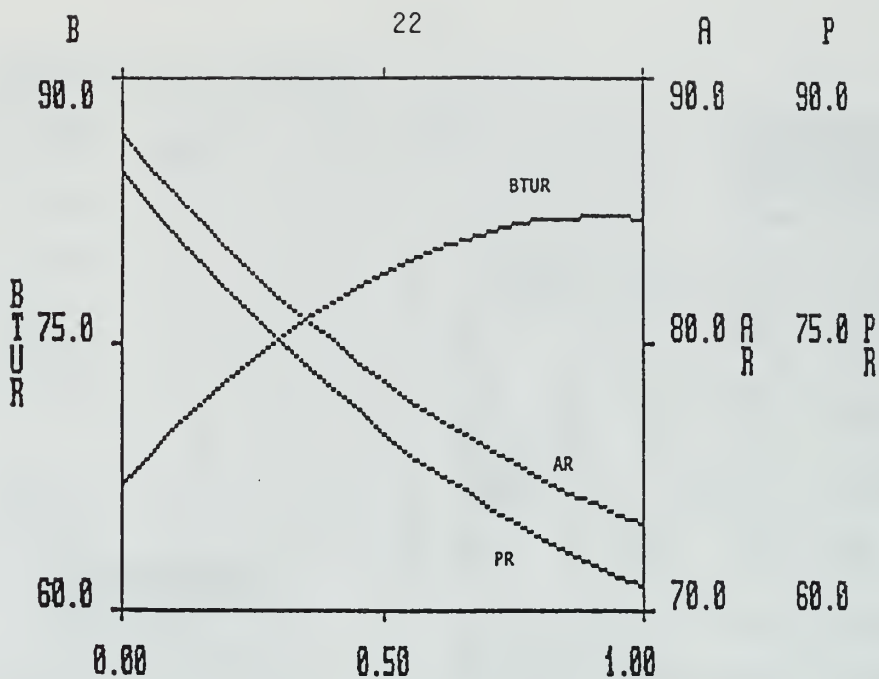


Figure 2

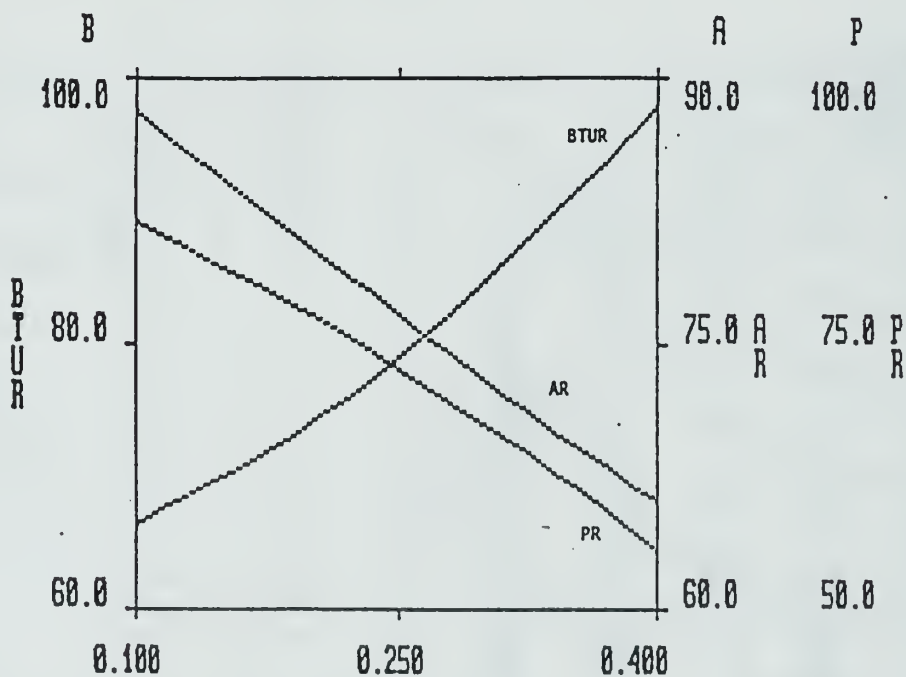


FRAC50

BTU Recovery, Ash and Pyrite Rejection as a Function of % 1008-11-50. Total dose is fixed at 0.25 lb active/tds (Upper Freeport ROM, 28x0)

TOTAL = 0.2500

Figure 4



TOTAL

BTU Recovery, Ash and Pyrite Rejection as a Function of Total Dose. lbs active/tds. Fraction of 1008-11-50 is constant at 50% (Upper Freeport Coal, 28x0)

FRAC50 = 0.5000

Examination of the wt% recovery versus time curves in Figure 6, indicates that for five of those surfactants tested and under the instrument conditions employed, over 70 wt.% is collected within 4 minutes and that over 85 wt.% is obtained within 6 minutes for four of these five reagents. This leads us to believe that the retention times within the continuous flow unit (four, 7-liter cells) should be no longer than ~ 10 minutes, with no less than 1 to 2 minutes/cell residence time. Aeration rate can be increased to increase the rate of flotation. However, many literature examples exist which indicate a lower ash and pyrite grade is obtained when low aeration rates are employed. Most of the reagents examined, with the exception of 1008-11-30, -31, approach the maximum recoveries asymptotically. Thus, increasing the overall retention time past a certain given time may improve recovery only incrementally. Often this last fraction is of higher ash and pyritic sulfur content. It may be advisable to "sacrifice" some Btu recovery and avoid returning excess mineral matter during a cleaner step. This could be of significance for those coals where there is potential for producing a compliance product (1.2 #SO₂/MM Btu) by pyritic sulfur removal.

Using Table 12 one can compare results for ash and pyrite rejection, as a function of either Btu recovery or time. From this data a new concept, "Surfactant-controlled flotation rate for ultrafine coal", is being developed. The basic tenets for control of recovery in froth flotation in the past have centered around interactions of frother dosage (generally MIBC or alkanolamide derivatives), collector dosage, aeration rate and recently for ultrafine flotation, bubble size. It is believed that an added factor now to be considered is that of "chemical" control. This data indicates that flotation "rate" can also be significantly controlled by specific surfactants.

In examining the across-the-board Btu recovery for equivalent times of collection (Table 12), recoveries differ by as much as a factor of two. Instead of designing circuitry for a specific retention time and recovery, one may determine what surfactant may be employed to achieve the optimum relationship between flotation rate and product quality. For existing circuitry, the same would apply and may not require extensive and/or expensive retrofits. There is current evidence that new cell designs are taking the retention time factor into account (Miller and Turbeville, 1986). The data also suggests that at equivalent Btu recovery, (although at differing times) resulting ash and pyrite rejections are approximately equal.

Electrokinetic Properties of Coals Used in the AF Process

Surface charges on the coal can be significantly influenced by the chemical environment of the coal/mineral/water system. To better understand the electrochemistry at the solid/liquid interface and the potential interaction between reagent and the solid/gas/liquid phases found in the process, electrokinetic studies have been initiated for two of the whole, washed coals used in AF testing. For the Illinois No. 5 (Springfield-SE) and Illinois No. 2 (Colchester-W) samples, the zeta potential curves of the whole coals in water and with various surfactant addition were determined (Figure 7-10). Surfactants studied included two non-ionic reagents (1008-11-26, 1008-11-31) and one anionic reagent (1008-11-39). For surfactants

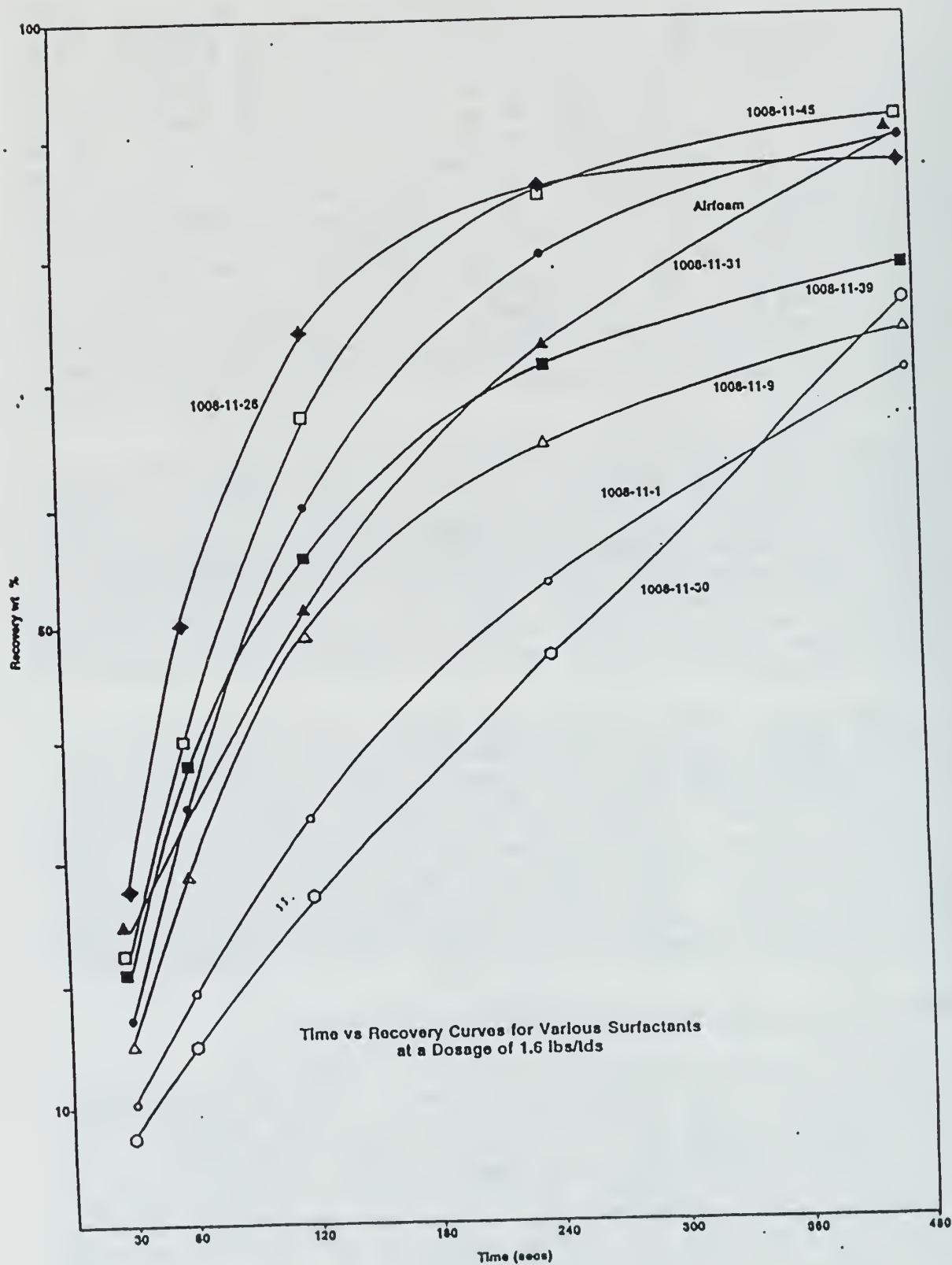


Figure 6.

Table 12 Timed Fractions for Recovery of Rougher Concentrates for Various Surfactants and the Resulting Ash, Pyrite Rejection and Btu Recovery (Illinois No.5, Springfield-SE).

SURFACTANT SERIES (1008-11-)									
Time, secs	1	9	26	30	31	39	44	15	
0-30	AR 96.8	92.8	87.9	97.4	91.9	90.0	91.0	91.45	
	PR 95.5	90.5	82.0	96.2	89.8	88.4	87.7	86.5	
	BtuR 10.1	17.0	31.0	9.0	25.0	21.8	21.2	22.2	
30-60	AR 93.2	85.2	78.5	95.1	88.4	81.2	81.7	82.6	
	PR 90.4	80.4	69.3	93.3	84.9	76.7	74.9	75.6	
	BtuR 21.7	34.0	54.2	17.5	36.2	40.9	42.7	43.5	
60-120	AR 88.9	80.7	65.8	91.3	81.9	71.7	68.8	68.9	
	PR 84.2	74.2	52.6	88.5	74.9	64.1	57.4	58.7	
	BtuR 36.5	44.0	79.4	30.5	56.3	60.8	69.8	72.3	
120-180	AR 84.9	72.0	59.4	88.2	77.0	66.9	61.3	60.5	
	PR 78.2	62.2	43.7	84.1	67.7	54.1	47.8	47.2	
	BtuR 48.7	64.0	88.7	40.9	69.3	71.2	83.4	86.2	
180-240	AR 79.6	70.0	55.4	84.3	73.1	64.0	56.3	56.0	
	PR 70.2	59.3	38.8	78.4	62.0	50.7	41.0	40.7	
	BtuR 58.2	69.0	92.8	52.1	78.1	76.8	90.6	91.8	
240-480	AR 71.7	65.7	51.7	68.9	58.7	58.7	47.7	49.1	
	PR 59.1	52.5	35.8	60.3	43.9	44.7	30.4	31.9	
	BtuR 76.7	79.0	94.1	83.4	96.2	85.1	95.5	97.0	

AR = Ash Rejection, PR = Pyrite Rejection, BtuR = Btu recovery

1008-11-31 and 1008-11-39, the zeta potential curves for two levels of surfactant addition were generated (Figures 9 and 10). These correspond to reagent concentrations that would result from reagent dosages of 1.2 and 2.4 lbs/ton at the standard conditions used for batch AF flotation testing. For 1008-11-26 the zeta potential curve was determined for only the highest dosage.

The zeta potential curve for the Illinois No. 5 (Springfield-SE) sample in water has a zero point of charge (zpc) near pH 4 which corresponds closely to coals of this rank as previously reported in the literature. The nonionic surfactants exhibit similar shaped curves to the coal in water but the zpc is shifted to near pH 5. This may point to the fact that the non-ionizing reagents, at these concentrations, function mainly as surface tension reduction agents and do not influence the charge on the coal surfaces. For the anionic surfactant (1008-11-39), the reversal of zeta potential for the portion of the curve to the left of the zpc is exhibited, along with a negative zeta potential through the entire pH range. This strongly suggests that the anionic surfactant is adsorbed onto the coal surface and apparently behaves as a potentially determining ion. Reagent 1008-11-39 is approximately as effective as 1008-11-26 and 1008-11-31 on a dosage/ recovery basis when performing AF tests, but was not as ash and pyritic sulfur rejective. This could be due to the surfactant being absorbed on mineral and gangue surfaces, thus acting as a collector which reduces selectivity. Judicial use of anionic surfactants are therefore required.

For the Illinois No. 2 (Colchester-W) sample the zeta potential of the whole coal in water was determined and compared to the zeta potential resulting from addition of surfactant 1008-11-39 (Figure 8). The zpc for the Illinois No. 2 (Colchester-W) sample in water was near pH 6. This value is quite high and unexpected and does not agree with previous literature references. This may possibly be due to using the whole coal as opposed to pure macerals. Coals of this rank should yield a zpc of pH 4; oxidation of the sample is also ruled out as the zpc would be shifted to lower values. Reagent 1008-11-39 again exhibited charge reversal in the region of positive zeta potential resulting in an overall negative zeta potential through the entire pH range. However, reagent 1008-11-39 was more effective than 1008-11-31 on a dosage/recovery basis when examined in AF testing.

Effectiveness of ISGS Aggregate Flotation as Compared to Alternate Micro-bubble Processes

Much emphasis has been placed recently on the significance of the relationship between bubble-size and flotation rate for ultrafine particles (Jameson, 1977; Yoon, 1984). While this may be one contributing factor for effective fine particle flotation, other properties such as specific surface area, surface charges, coal type, and interactions with the reagents, may equally contribute to the successful flotation of ultrafine coal. New cell designs which incorporate the microbubble process have also appeared recently.

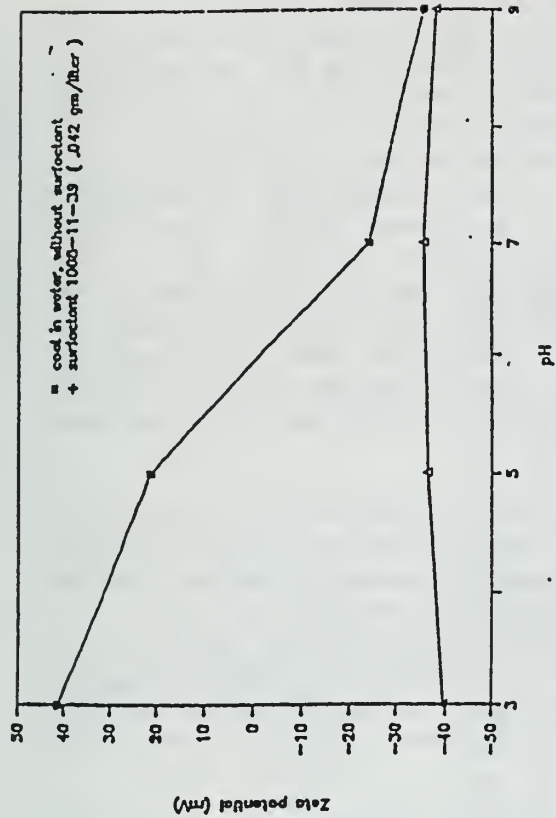


Fig 8 Zeta potential in water and with surfactant 1008-11-39 (Colchester-W sample)

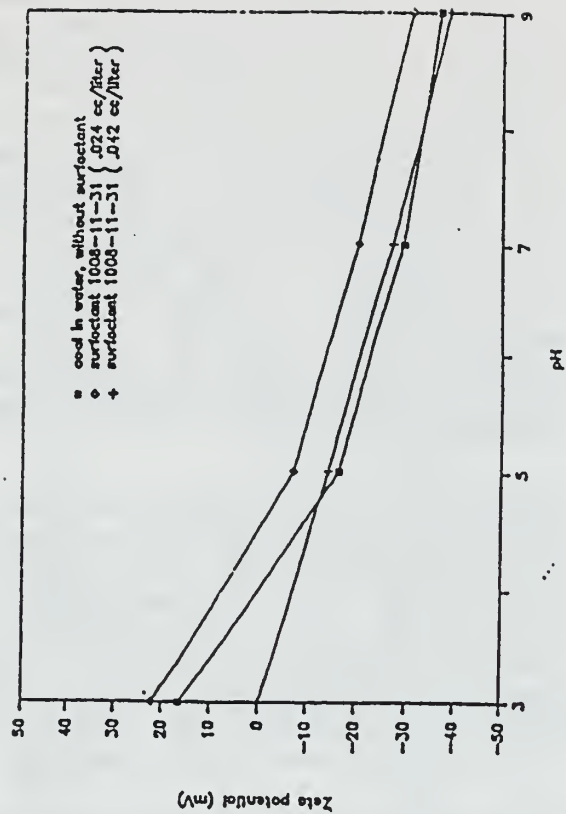


Fig 10 Zeta potential in water and with two levels of surfactant 1008-11-31 dosage (Springfield-SE sample)

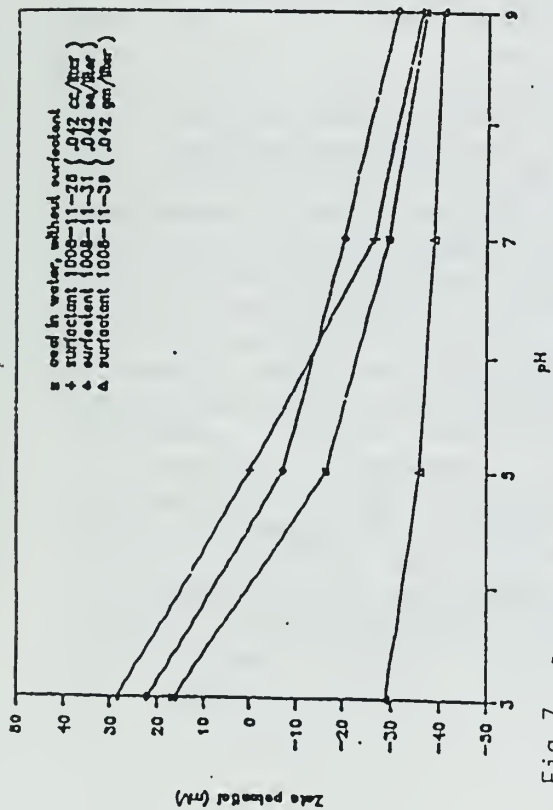


Fig 7 Zeta potential in water and with surfactant addition (Springfield-SE sample)

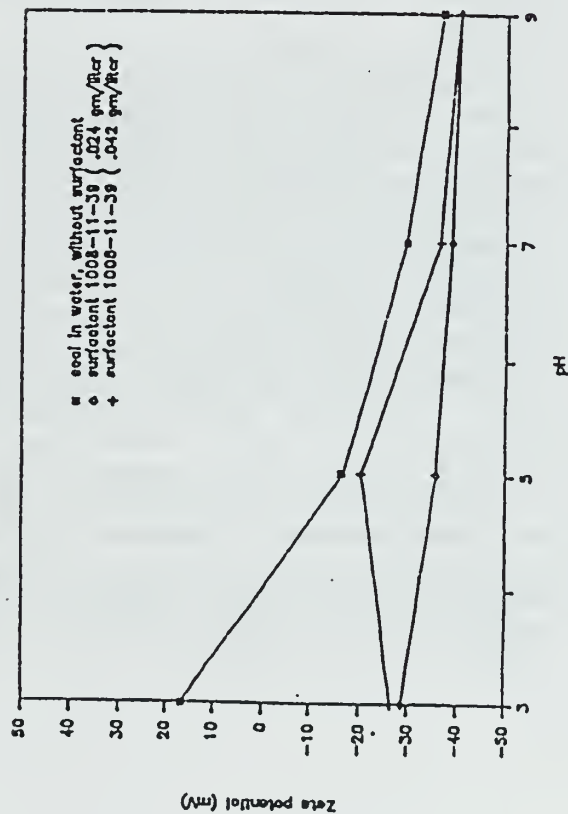


Fig 9 Zeta potential in water and with two levels of surfactant 1008-11-39 dosage (Springfield-SE sample)

To examine flotation cell differences, experiments were conducted on two different types of flotation units. One, the Denver D-2 sub-aeration cell, produces a dispersion of rather coarse bubbles utilizing air pulled into the cell by the rotating impeller. Air bubbles are sheared and dispersed throughout the cell by the action of the impeller/diffuser mechanism. The other cell employed was produced by a manufacturer (Wilfley-Weber) whose unit design incorporates the concept of "in-situ" microbubble generation. This is accomplished by forcing air through a porous ceramic frit (4-8 micron), with shearing/mixing of the bubbles being achieved by a low profile impeller. With such a wide divergence of bubble size produced in these two types of units, it was anticipated that results should clearly indicate bubble size and equipment effects. (Results from the previous contract period (Read et. al, 1986) indicated cell design may not be as critical as previously thought. These results are presented again in the following discussion.)

The operating conditions were held constant in both cells (4L, 8scfh air, 1075rpm, 3.5%-4.5% solids) and the Illinois No. 5 (Springfield-SE) washed coal (80% -400 mesh) was subjected to dosage/recovery testing while employing the 1008-11-44 surfactant. The summarized results in Table 13, for recovery and ash/pyrite rejection, reveal unexpectedly that under identical operating conditions and reagent dosages no experimentally discernable differences exists. (There are some instances where pyrite rejection is higher, but this is attributed to mass balance errors and limitations in analytical accuracy).

Additional evidence obtained photographically showed the bubble size in the two cells to be essentially the same (100-500 microns) at the equivalent surfactant loading. The initial thought that this size control was a function of this one particular surfactant was negated by testing of an alternate surfactant, 1008-11-31, which showed essentially the same results.

The tentative conclusion is that the "initial" bubble size generated by either unit is not the controlling factor for fine bubble generation. The results indicate that "final" bubble size may be a function of the reagent and its influence on factors such as surface tension, bubble growth and coalescence. This concept appears to be further supported by work conducted on coalescence of gas bubbles which employed both inorganic electrolytes and organic, non-ionizing alcohols (Marrucci and Nicodemo, 1967). Gas bubbles generated in a column cell with a porous plate (8 micron) were found to have a constant mean size for different gas flow rates in aqueous solutions, with the coalescence process taking place near the distributor plate. Addition of an electrolyte, or non-ionizing alcohol, prevented coalescence and with increasing concentration, produced a minimum attainable bubble size. Chemical control of bubble size, which is a function of preventing coalescence, is therefore plausible. Additional research using the concept of chemical control of bubble size for flotation of fine and ultrafine coal (Hansen and Klimpel, 1987; Panopolous et al, 1986, Bustamonte, 1985) further supports ISGS conclusions regarding reagent control versus "new" cell design.

Table 13 Comparison of Recovery and Ash, Pyrite Rejection for the Wilfley-Weber and Denver Flotation Cells

<u>Reagent</u>	<u>Dosage</u> ¹		<u>Wilfley-Weber</u>	<u>Denver</u>
1008-11-		WT%	58.3	58.9
		Ash Rej	78.1	76.2
-44	1.2	Pyr Rej	74.7 ²	65.8
		Btu Rec	63.7	65.1
		WT%	84.9	86.2
		Ash Rej	62.8	58.2
-44	1.4	Pyr Rej	45.3	41.5
		Btu Rec	91.4	93.9
		WT%	93.1	90.1
		Ash Rej	37.4 ²	47.7
-44	1.6	Pyr Rej	21.6 ²	30.4
		Btu Rec	98.5	95.6
		WT%	87.6	90.1
		Ash Rej	60.2	62.4
-31	1.6	Pyr Rej	46.7	49.1
		Btu Rec	94.2	96.8

¹ Dosage in lbs active/ton dry solids

² Ash and pyrite rejection differences attributed to mass balance errors.

The influence of the reagent on bubble size and the resulting surface chemistry may control not only the bubble size, but the chemical (chemisorptive) interaction of the reagent with the coal surface as well. While this finding does not dismiss the role of microbubble size effects, or cell hydrodynamics, it does suggest stronger interactions between the surface chemistry of the coal and reagents than have been reported in past studies and that flotation equipment specifically designed to generate microbubbles may not have any advantage over conventional cells if selective reagents are employed. Further confirmation of these conclusions were obtained by comparing results to an alternate flotation unit (Deister Flotaire) which generates "external" microbubbles prior to introduction into the cell. Such a test was arranged by CRSC, and results are discussed in the following section.

Effectiveness of ISGS Aggregate Flotation as Compared to UCC Column (Microbubble) Flotation

A comparative study was arranged by CRSC to determine whether AF could be utilized for producing ultraclean coal and to compare results of AF to that of UCC Column Flotation. Results for both tasks are discussed in further detail below.

Production of Ultra Clean Coal with < 2% Ash by ISGS Aggregate Flotation

While the current work has focused on reducing pyritic sulfur and ash, an interest was expressed as to whether the AF process is capable of producing feedstock suitable for coal-water-slurry (CWS). Samples of coal from the Cedar Grove Seam (VA) were obtained from United Coal Company which is currently producing CWS from this feed after cleaning by means of microbubble column flotation.

Analyses for the Cedar Grove ROM sample showed it to be very clean; 3.0% ash, 0.56% total sulfur and 14775 Btu. Results for the four minute stirred ball mill grind (1000g charge, 33% solids by weight) appear in Table 14. This treatment yields 80% passing 400 mesh, with 86% of the ash being distributed in this single fraction. This is the typical particle size feed employed in AF.

The initial AF result for this sample with timed collection of the rougher concentrate at a dosage of 1.0 lb/ton of reagent (1008-11-31), is shown in Table 15. It is apparent that the Cedar Grove sample floats faster than typical Illinois Basin coals yielding 62% Btu recovery in 2 minutes and 82.8% Btu recovery within 4 minutes. The cumulative results indicate that at 8 minutes the Btu recovery for AF is 90% and a product with cumulative ash and total sulfur of 0.96% and 0.54%, respectively are obtained. This test indicated near optimal recovery for the particular reagent used under the constraint given for product quality.

Additional flotation tests and results with the 1008-11-26 reagent are given in Table 16. The results for the first 4 minute collection period (Concentrate 1) show a product with 87% Btu recovery, 1% ash and 0.54% total sulfur. With this data, it appears that suitable feeds for the production of CWS may be attained with the AF process.

Comparison of Reduction of Ash and Sulfur in Illinois Coals by UCC Column Flotation

A 50 lb sample of the Illinois No. 5 (Springfield-SE) ROM coal was obtained by UCC from ISGS in order to study the effectiveness of Column Flotation in reducing ash and sulfur in Illinois coal. Both single and two stage column flotation tests were conducted. For the first stage, approximately 10% solids of 100 mesh x 0 slurry was fed into the UCCRC column flotation cell, and its froth product was re-cleaned through the column cell after re-grinding it to 400 mesh x 0.

The test results are as shown in Tables 17 and 18. Through the first stage, the reduction of ash and sulfur was 49.5% and 46.0%, respectively, with 82.7% combustible recovery. However, the froth product ash and sulfur was fairly high (19.88% ash and 2.97% sulfur). For the second stage cleaning step, the reduction of ash and sulfur was 87.2% and 77.7%, respectively, with an overall recovery of 46.2%. The froth product ash and sulfur showed 10.11% and 2.46%, respectively.

Comparative data as obtained by AF for a number of reagents on the same coal sample are given in Table 19. For further comparative information, a rougher/cleaner AF separation is given in Table 20. Comparison of the two

Table 14 Particle Size Distribution for Cedar Grove Coal after 4 Minute Stirred Ball Mill Grind.

Mesh (US)	Elementary %			Cumulative, %			Distribution, %		
	Wt	Ash	TS	Btu	Wt	Ash	TS	Btu	Btu
+200	2.31	4.30	.66	14928	2.31	4.30	.66	14928	2.34
200x270	5.68	1.80	.55	14994	7.99	2.52	.58	14975	5.77
270x325	5.73	1.80	.58	14977	13.72	2.22	.58	14976	5.82
325x400	6.28	1.80	.56	15028	20.00	2.09	.57	14992	6.39
-400	80.00	3.40	.56	14692	100.00	3.14	.56	14752	79.66

Table 15 Aggregate Flotation Results for Cedar Grove Coal (1.0 lb/tds 1008-11-31).

	Elementary %			Cumulative %		
	Wt	Ash	TS	Btu	Wt	Ash
Con-1	19.14	.90	.58	15071	19.14	.90
Con-2	17.79	.90	.55	14997	36.93	.89
Con-3	12.93	.90	.55	15076	49.86	.90
Con-4	10.89	.90	.54	15087	60.75	.90
Con-5	13.43	.90	.54	15093	74.18	.90
Con-6	6.71	1.40	.52	15077	80.89	.94
Con-7	7.92	1.10	.53	15007	88.81	.96
TAILS	11.19	19.30	.63	11986	100.00	3.01

Table 16 Aggregate Flotation Results for Cedar Grove Coal (1.0 lb/tds 1008-11-26).

	Elementary %			Cumulative %		
	Wt	Ash	TS	Btu	Wt	Ash
Con-	85.54	1.00	.54	15023	85.54	1.00
Con-2	6.03	2.00	.57	14968	91.57	1.06
Tails	8.43	25.60	.63	10819	100.00	3.14

Btu
15023
15019
14665

Table 17 . Two Stage Column Flotation Tests for the Illinois No. 5 (Springfield-SE) Coal.

A. First stage with raw coal (100 mesh x 0)

	<u>Wt. (%)</u>	<u>Ash (%)</u>	<u>Sulf. (%)</u>
Froth	73.4	19.88	2.97
Tailings	26.6	53.67	6.99
Total	100.0	28.87	4.04

Note: Feed material was conditioned with 6 drops of Diesel oil for 2 minutes before flotation. The reagent used was 10% solution of Aero 65.

B. Second Stage with the froth of first stage (400 mesh x 0)

	<u>Wt. (%)</u>	<u>Ash (%)</u>	<u>Sulf. (%)</u>
Froth	49.8	10.11	2.46
Tailings	50.2	29.57	3.48
Total	100.0	19.88	2.97

Note: The reagent was 10% solution of Aero 65.

Table 18. Ash and Sulfur Distribution in the Products of Two Stage Column Flotation for the Illinois No. 5 (Springfield-SE) Coal.

	<u>Wt. (%)</u>	<u>Recovery (%)</u>	<u>Ash Distr. (%)</u>	<u>Sulf. Distr. (%)</u>
2nd Stage Froth	36.6	46.2	12.8	22.3
2nd Stage Tailings	36.8	36.5	37.7	31.7
1st Stage Froth	73.4	82.7	50.5	54.0
1st Stage Tailings	26.6	17.3	49.5	46.0
Head Sample	100.0	100.0	100.0	100.0

Table 19. Single Stage AF Results for Illinois No. 5 (Springfield-SE) ROM Coal.

Reagent	Dosage	BtuR	Product, %			Rejection, %		
			Ash	TS	PS	Ash	TS	PS
1008-11-31	1.6	77.8	5.6	2.6	0.82	89.1	62.8	82.7
	1.7	89.3	6.3	2.7	0.92	85.3	53.9	76.4
	2.0	96.4	8.6	3.0	1.16	79.6	47.6	69.0
1008-11-26	1.6	85.4	5.9	2.7	0.79	87.3	57.0	80.8
	2.2	94.0	8.0	2.9	1.12	85.1	55.1	75.5
1008-11-45	1.3	55.7	7.3	2.6	0.75	89.5	70.8	86.3
	1.5	80.0	9.0	3.0	1.19	83.0	56.7	74.0

Table 20. Dual Stage AF Results for Illinois No. 5 (Springfield-SE) ROM Coal.

Reagent	Total Dosage	Total time of Collection	BtuR	Product, %			Rejection, %		
				Ash	TS	PS	Ash	TS	PS
1008-11-31	2.8	0-240	79.3	4.8	2.6	0.67	91.1	63.3	85.3
		0-420	93.4	5.9	2.7	0.81	87.0	54.3	78.9
1008-11-26	2.9	0-240	91.1	5.6	2.8	0.88	88.0	56.6	79.1
		0-420	98.2	6.87	3.0	1.12	84.1	48.8	70.9

Table 21. Comparison of Column Flotation and AF Results for the Illinois No. 5 (Springfield-SE) Coal.

	UCC Column Flotation	ISGS AF
<u>Single Stage</u>		
Recovery	82.7 ¹	85.4 ²
Ash Rejection	49.5	87.3
Total Sulfur Rejection	46.0	57.0
Pyritic S Rejection	---	80.8
Product Ash	19.8	5.9
Product TS	3.0	2.7
Product PS	---	0.79
<u>Dual Stage</u>		
Recovery	46.2 ¹	79.3 ²
Ash Rejection	87.2	91.1
Total S Rejection	---	85.3
Product, Ash	10.1	4.8
Product TS	2.5	2.6
Product PS	---	0.67

¹Combustible recovery²Btu recovery

different processes for given recoveries are shown in Table 21. For the single stage tests and near equivalent recovery (85%), the AF process shows a significantly superior degree of ash rejection (Column=49.5%, AF = 87.3%) with better total sulfur rejection and lower product ash and total sulfur. For the dual stage test, results are difficult to compare at equal recovery due to low values obtained by column flotation. Nevertheless it is quite obvious that the AF process yields a higher Btu recovery (almost two fold greater) with high ash and total sulfur rejection. Again, product quality is better from AF. These results suggest that AF is a superior method as compared to column flotation for cleaning the ultrafine Illinois No. 5 (Springfield-Se) coal.

Characterization of the Mineral Matter Liberation Phenomena

Procedures and preliminary tests for determining the optimum relationships between particle size reduction and ash and pyrite rejection as obtained by AF were initiated for the Illinois No. 5 (Springfield-SE) sample. A rod mill (7 1/2 I.D. x 9 1/2" long) is being used to prepare size distributions ranging from 80% passing 65 mesh to 80% passing 200 mesh. Rods were chosen as grinding media over balls because a narrower size distribution can be produced like that generated by a ball mill in closed circuit with a classifying device. The grinding media for the rod mill consists of an assortment of rods ranging from 1/2" to 1" with the total weight of grinding media being 32.1 lbs. The mill is operated at 62% of critical speed which is in the range recommended for rod mills (60-68%).

Petrographic analysis has been used to determine the relationship between pyrite and mineral matter liberation and particle size distribution. This information will be used in conjunction with flotation data at these size distributions to devise an economically efficient grinding scheme. The relationship between reagent consumption/rejection and particle size distribution will also be investigated. Preliminary results are further discussed in the Petrography section.

Production of Compliance (1.2#SO₂/MMBtu) Illinois Basin Coal

A significant reserve of approximately 3.5 billion tons of low organic, high pyritic sulfur Illinois coals exists (Harvey, 1986) which has significant market potential (Fonseca, 1986) if physically cleaned to compliance sulfur levels. Studies on this aspect, conducted both at ISGS and Northwestern, have proven that deep physical cleaning is a viable method for one particular coal from the Illinois No. 6 (Herrin-SC2) seam. ISGS batch results in Tables 22 and 23 indicate that a compliance product can be made while maintaining relatively high Btu recovery (>80% overall based on ROM feed). Preliminary results at ISGS (Tables 24-26) have further indicated that Btu recoveries of 92%-95% can be attained on a precleaned feed while maintaining the compliance goal. Further tests are anticipated for this sample during the next year. It was also initially observed during evaluation of this high chlorine coal (0.35 wt.% ROM), that ultrafine grinding reduced the chlorine content to 0.15 wt. %, a reduction of approximately 60%. The chlorine reduction aspect, as a function of particle size, will also be further explored during the next contract period.

Table 22 Results for Illinois No. 6 (Herrin-SC2) ROM Coal by Rougher/Cleaner Aggregate Flotation

Collection Time, sec	Elementary, %					Cumulative, %					#SO ₂ /MM BTU			
	WT	Ash	PS	TS	BTU	WT	Ash	PS	TS	BTU	BTUR	PR	AR	
Conc 1	0-120	27.63	3.3	0.20	0.77	14160	27.63	3.3	0.20	0.77	14160	36.8	91.7	96.4
2	120-240	16.10	3.7	0.26	0.79	14085	43.73	3.5	0.22	0.78	14130	58.3	85.4	94.1
3	240-360	8.89	4.7	0.31	0.84	13875	52.62	3.6	0.24	0.79	14085	69.9	81.2	92.4
4	360-480	4.90	6.5	0.41	0.93	13683	57.52	3.9	0.25	0.80	14050	76.2	78.2	91.2
														1.14
Cleaner Tails	---	6.77	29.8	1.06	1.33	8835	64.29	6.6	0.34	0.86	13500	81.9	67.4	83.2
														1.27
Rougher Tails	---	35.72	59.1	1.25	1.35	5385	100.0	25.4	0.66	1.03	10600			1.94

1.94

BTUR = % BTU Recovery, PR = % Pyritic Sulfur Rejection, AR = % Ash Rejection; 4.3 lb/ton Rougher, 1.9 lb/ton Cleaner 1008-11-31

Table 23 Results for Illinois No. 6 (Herrin-SC2) ROM Coal by Rougher/Cleaner Aggregate Flotation

Collection Time, sec	Elementary, %				Cumulative, %				#SO ₂ /MM						
	WT	Ash	PS	TS	BTU	WT	Ash	PS	TS	BTU	BTUR	PR	AR	BTU	
Conc 1	0-120	27.6	3.3	0.21	0.78	14050	27.6	3.3	0.21	0.78	14050	36.7	91.3	96.4	1.11
2	120-240	14.3	3.6	0.23	0.79	13950	41.9	3.4	0.23	0.78	14010	55.4	85.6	94.4	1.11
3	240-480	13.4	5.1	0.26	0.88	13750	55.3	3.8	0.26	0.81	13950	73.1	78.3	91.7	1.16
Cleaner Tails	14.0	16.7	0.34	1.08	11990	69.3	6.4	0.34	0.86	13550	89.0	64.7	82.5	1.26	
Rougher Tails	30.7	68.1	1.39	1.39	3770	100.0	25.4	0.66	1.02	10540				1.93	

1.11
1.11
1.16
1.26
1.93

BTUR = % BTU Recovery, PR = % Pyritic Sulfur Rejection, AR = % Ash Rejection, Rougher: 2.0 lb/t -31, 1.0 lb/t -51; Cleaner: 1.0 lb/t -31, 0.4 lb/t -51

Table 24 Results of Rougher Only Aggregate Flotation for the Illinois No. 6 (Herrin-SC2) Clean Coal Product

Fig. 24 Results of Rougher Only Aggregate Flotation for the 100-mesh material																	
Collection Time, sec	Elementary, %					Cumulative, %					Recovery, %		Rejection, %		#SO ₂ /MMBTU		
	WT	Ash	PS	OS	TS	WT	Ash	PS	OS	TS	BTU	Cl	Ash	Pyrite			
1 0-240	73.3	3.5	0.24	0.56	0.80	14060	0.16	0.24	0.56	0.80	14060	0.16	77.8	70.4	60.3	1.14	
2 240-480	16.4	6.1	0.40	0.54	0.94	13740	0.16	4.0	0.27	0.54	0.83	14000	0.16	94.8	58.8	45.4	1.19
3 480-720	10.3	49.7	1.96	0.27	2.23	6651	0.07	100	8.7	0.44	0.53	0.97	13245	0.15	---	---	1.46

Agent: 1008-11-31; 5.0 lb/t; 4 l Denver cell, 1075 rpm, 8 scfh; Note: ROM feed analyzed 0.35 wt% Cl. Ultrafine grinding by 5 minute wet Stirred Mill reduced (soluble) chlorine to 0.14 wt%.

Table 25 Results of Rougher/Cleaner Aggregate Flotation for the Illinois No. 6 (Herrin-SC2) Clean Coal Product

Table 25 Results of Rougher/Cleaner Aggregate Flotation for the Illinois No. 0 (Net in SO ₂) Cream Feed																
Collection Time, sec	Elementary, %					Cumulative, %					Recovery, %		Rejection, %		#SO ₂ /MMBTU	
	WT	Ash	PS	OS	TS	WT	Ash	PS	OS	TS	BTU	BTU	Ash	Pyrite		
C 1	42.0	2.4	0.20	0.56	0.77	14150	42.0	2.4	0.20	0.56	0.77	14150	45.3	88.6	81.2	1.08
C 2	27.2	3.4	0.23	0.57	0.79	14035	69.2	2.87	0.21	0.56	0.80	14110	74.4	78.2	67.3	1.13
C 3	14.9	5.0	0.34	0.54	0.80	13670	84.1	3.2	0.23	0.56	0.80	14030	89.9	69.8	60.0	1.14
aner Tails	3.3	22.1	1.25	0.45	0.84	10920	87.4	3.9	0.27	0.56	0.84	13915	92.6	61.6	46.8	1.21
ther Tails	12.6	43.4	1.67	0.29	0.98	7720	100	8.9	0.45	0.52	0.98	13135				1.49

Agent: 1008-11-31; Rougher = 5.5 lb/t, Cleaner = 2.7 lb/t; 4 l Denver cell, 1075 rpm, 8 scfh; 5 minute Stirred Ball Mill Grind.

Table 26 Results of Staged Reagent Addition Aggregate Flotation for the Illinois No. 6 (Herrin-SC2) Clean Coal Product

Dosage ul	Elementary, %					Cumulative, %					Recovery, %		Rejection, %		#SO ₂ /MMBTU	
	WT	Ash	PS	OS	TS	BTU	WT	Ash	PS	OS	TS	BTU	BTU	Ash		Pyrite
1 160	7.1	3.1	0.22	0.55	0.77	13930	7.1	3.1	0.22	0.55	0.77	13930	7.5	97.6	96.7	1.10
2 80	13.7	3.1	0.22	0.55	0.77	14000	20.8	3.1	0.22	0.55	0.77	13970	22.2	93.1	90.2	1.10
3 80	22.8	3.2	0.23	0.57	0.80	14063	43.6	3.2	0.23	0.56	0.79	14020	46.6	85.2	78.9	1.13
4 80	28.9	4.3	0.28	0.57	0.85	13905	72.5	3.6	0.25	0.56	0.81	13975	77.3	71.8	61.6	1.16
5 80	17.4	7.1	0.42	0.54	0.95	13451	89.9	4.3	0.28	0.56	0.84	13875	95.2	58.5	45.9	1.21
15 80	10.2	53.3	2.10	0.15	2.25	6150	100	9.3	0.47	0.52	0.98	13090	---	---	---	1.50

Agent: 1008-11-31; Total Dose = 6.3 lb/t; Denver 4 l cell, 1075 rpm, 8 scfh; 5 minute Stirred Ball Mill Grind

Influence of Circuitry Design

To compare effectiveness of rougher only versus rougher/cleaner flotation, test results using several of the most cost-effective reagents (-26, -31, -44) for both the washed and ROM Illinois No. 5 (Springfield-SE) coal, are given in Tables 27 and 28.

The overall improvement appears to be approximately 5%-8% in both ash and pyrite rejection at equivalent Btu recovery when utilizing a rougher/cleaner cycle for the ROM coal. For example, there should be a corresponding decrease in ash and pyrite rejection with increasing Btu recovery. The rougher/cleaner cycle falls outside this pattern for either reagent tested. The washed sample also shows a similar behavior yet the overall net increase in improved ash rejection is only 3%-5%. The pyrite rejection values for the tabled sample show virtually no change for the rougher/cleaner cycle.

The concentrate grades also appear to be slightly improved with a rougher/cleaner cycle, but this may be a reflection of the feed grade and degree of Btu recovery. Nevertheless, results for the ROM sample at 93.4 and 96.4% Btu recovery (1008-11-31) do show a lower ash and pyrite grade for essentially equal feed grades in the rougher/cleaner versus rougher. For the case of 98.2 (1008-11-26) and 96.4 (1008-11-31) %Btu recovery, a similar result for ash grade is obtained, but pyrite grade is equivalent for rougher versus rougher/cleaner. This last example may be a result of the reagents employed.

It is also clear that some increase in reagent dosage, on the order of 0.2 to 0.3 lbs/ton feed, is required to produce equivalent Btu recovery for the ROM feed versus tabled product for a rougher stage only. Reagent consumption for a rougher/cleaner cycle adds an additional 0.5 to 1.0 lbs/ton feed to that required for rougher only. It may be that the costs of additional reagent will make a rougher/cleaner cycle uneconomical unless the feedstock is ROM or possibly impounded waste fines with a high ash content. Additional Btu recovery would also need to be balanced by increased grinding cost of ROM and additional cell volume requirements. In a conventional AF plant, one could expect processes such as jigging, or cycloning to remove coarse refuse prior to ultrafine grinding/flotation.

If the overall Btu recovery for the Tabling/AF step is 80%, one should compare these results to 80% Btu recovery for the ROM feed. Comparison of such data from Tables 27 and 28 gives a product grade having 4.8% ash and 0.67% pyritic sulfur, at 91.1% and 85.3% ash and pyrite rejection, respectively. The combined tabling/AF step, at an overall 80% Btu recovery, would produce a product with similar grade (4.6% ash and 0.68% pyritic sulfur) and an overall ash and pyrite rejection of approximately 88% and 85%.

Recoveries expressed in portions of the previous discussion are based on the flotation step for the washed coal. A Btu recovery for the pre-treatment wash of 80%-85% would be accompanied by an overall net Btu recovery for the tabling/AF process of 75%-80%. In past efforts, internal gravity separation processes were applied in order to maximize rejection, often at the expense of some Btu recovery. Current methodology now used is aimed more towards Btu recovery, with recent tests yielding 90%-93% from

Table 27 Comparison of Rougher and Rougher/Cleaner AF for the Illinois No. 5 (Springfield-SE) Washed Coal

Reagent	Dosage	Stage	Collection Time, Sec	Btu Rec	Feed %			Product %			Rejection, %	
					Ash	Pyrite	Ash	Pyrite	Ash	Pyrite	Ash	Pyrite
1008-11-26	1.78	R/C	0-240	93.9	9.8	0.92	3.9	0.57	65.4	45.8		
1008-11-26	1.40	R	0-480	96.9	11.3	1.15	5.3	0.73	57.7	42.7		
1008-11-26	1.78	R/C	0-480	98.4	9.8	0.92	4.3	0.58	59.9	41.9		
1008-11-31	2.55	R/C	0-240	73.4	9.6	1.01	3.5	0.49	74.9	66.9		
1008-11-31	1.40	R	0-480	74.3	11.2	1.02	4.6	0.56	71.7	62.2		
1008-11-31	2.55	R/C	0-480	91.7	9.6	1.01	4.0	0.57	63.7	52.0		
1008-11-31	1.60	R	0-480	96.8	11.1	1.05	5.1	0.65	58.6	43.3		
1008-11-44	1.20	R	0-480	65.1	11.9	1.20	4.8	0.69	76.2	65.8		
1008-11-44	2.05	R/C	0-240	85.8	9.7	1.01	4.6	0.68	61.9	45.5		
1008-11-44	2.05	R/C	0-480	91.8	9.7	1.01	4.7	0.70	58.2	40.0		
1008-11-44	1.40	R	0-480	93.9	12.2	1.16	5.9	0.78	58.3	41.7		

Dosage in lbs active/ton dry feed; R=Rougher only, R/C=Rougher, Cleaner; Feed Ash and Pyrite differences due to new splits of washed coal;

Table 28 Comparison of Rougher and Rougher/Cleaner AF for the Illinois No. 5 (Springfield-SE) ROM Coal

Reagent	Dosage	Stage	Collection Time, Sec	Btu Rec	Feed %			Product %			Rejection, %	
					Ash	Pyrite	Ash	Pyrite	Ash	Pyrite	Ash	Pyrite
1008-11-26	1.55	R	0-480	85.4	28.9	2.55	5.9	0.79	87.3	80.8		
1008-11-26	2.90	R/C	0-240	91.1	30.2	2.73	5.6	0.88	88.0	79.1		
1008-11-26	2.20	R	0-480	94.0	34.6	3.02	8.0	1.12	85.0	76.2		
1008-11-26	2.90	R/C	0-480	98.2	30.2	2.73	6.8	1.12	84.1	70.9		
1008-11-31	1.64	R	0-480	78.6	29.3	2.71	5.6	0.87	88.9	81.7		
1008-11-31	2.78	R/C	0-240	79.3	30.2	2.56	4.8	0.67	91.1	85.3		
1008-11-31	1.72	R	0-480	89.0	28.4	2.57	6.3	0.92	85.3	76.4		
1008-11-31	2.78	R/C	0-480	93.4	30.2	2.56	5.9	0.81	87.0	78.9		
1008-11-31	2.03	R	0-480	96.4	30.1	2.67	8.6	1.16	79.6	69.0		

Dosage in lbs active/ton dry feed; R=Rougher only, R/C=Rougher, Cleaner
AR = % Ash Rejection, Pr = % Pyritic Sulfur Rejection

the tabling step. Future work in gravity separation/flotation is expected to yield a higher net Btu recovery of 85% or greater.

Recovery of Coal from Prep Plant Waste Streams

While the AF process has been predominately geared toward examination of ROM and precleaned feeds, there is the potential that it may also serve as an enhanced coal recovery process for existing fine refuse impoundment ponds and coarse gob piles. To assess this possibility evaluations have been performed on both refuse stream feeds and gob pile material obtained from various preparation plants.

Results from both multiple stage froth flotation and AF for refuse streams produced concentrates of saleable product quality. While the froth flotation product was slightly lower in ash (5.0% vs 6.9%), the AF process retained a much higher Btu recovery (82% vs 52%). This was quite encouraging considering that this feed is currently being disposed of due to poor filtration characteristics as a result of high clay content (ash).

Work on gob pile material proved to be more difficult (48.7% ash, 7.1% Total S). Initial work-up included desliming and crushing to 100% -6 mesh followed by tabling of this fraction which yielded a concentrate with 27.8% ash, 4.02% total S and 64% Btu recovery. The concentrate was then ground to yield 80% -400 mesh and treated by the AF process. The final product contained 12.2% ash, 3.2% total S with 51% Btu recovery.

Several other tests have been performed during the current contract on refuse tailings (-100 mesh fractions). Using single stage AF tests with no additional grinding, a feed with 58.9% ash, 1.75% PS, 2.37% TS and 5135 Btu/lb yielded a product of 14.7% ash, 1.3% PS, 2.82% TS and 12160 Btu/lb at an overall 93.7% Btu recovery (Table 29). Reagent requirements at 5.8 lbs/ton were high, but could be due to the additional clay/shale material present which adsorbs such reagents.

Other work on actual -100 mesh froth flotation feed (33.1% ash, 1.5% PS, 1.94% TS, 9590 Btu) has been very encouraging. Results in Table 30 indicate that good product quality can be maintained with high Btu recovery when employing selective reagents used in the AF process, even without additional ultrafine grinding. One notable aspect for this particular feed is that if ultrafine grinding had been employed, reduction in the pyritic sulfur may have led to production of a compliance product. Efforts for this feed using this approach are continuing.

The conclusion from the work for the plant waste materials and -100 mesh flotation feed is that Aggregate Flotation can be used to recover valuable product from the coarse gob piles and fine plant refuse streams. However, the plant outwash material is easier to upgrade than the coarse gob pile material and appears to be a function of liberation. The gob pile material also consisted of a great deal of coal which contained shale partings. In the future, confirmation by additional batch tests could lead to evaluations in the continuous flow unit.

Table 29 AF Results for -100 Mesh Refuse Stream

	<u>WT</u>	<u>Btu</u>	<u>Elementary, %</u>			<u>TS</u>	<u>Btu/lb</u>
			<u>Ash</u>	<u>PS</u>	<u>OS</u>		
Concentrates	39.6	93.7	14.70	1.30	1.52	2.82	12160
Tails	60.4	6.2	87.9	2.04	0.01	2.07	531
Calculated Feed	100.0	100.0	58.92	1.75	0.61	2.67	5135

Reagent: 1008-11-31, 5.8 lbs active/ton dry solids

Table 30 AF Results for -100 Mesh Froth Flotation Feed

<u>Concentrate</u>	<u>WT</u>	<u>Btu</u>	<u>Cummulative, %</u>			<u>TS</u>	<u>Btu/lb</u>
			<u>Ash</u>	<u>PS</u>	<u>OS</u>		
1 (0-1 min)	46.2	68.2	5.8	0.52	0.66	1.19	14160
2 (1-2 min)	58.8	86.1	6.6	0.60	0.62	1.24	14050
3 (2-4 min)	66.6	95.6	8.2	0.78	0.45	1.40	13785
Tails	100.0	100.0	33.1	1.50	0.44	1.94	9590

Reagent: 1008-11-31, 1.1 lbs active/ton dry solids

Options for Aggregate Flotation Final Product Form

As problems associated with shipping and handling of fines are well documented, a viable final AF product form must be established. It is felt that this form may be limited to two options, pelletization or coal-water-slurry (CWS), with utilization being dictated by end-user specifications. Coal pelletization is a well-known and accepted technology, and has been proven on an industrial scale, but CWS may require an actual market before this product option can be exercised. A third possibility does exist, the ultrafine coal cleaning plant could be located on-site, or adjacent to, existing coal-fired electrical generating plants. In this case, both pelletization or slurry forms would be unnecessary, as the dry product (-400 mesh) could be pneumatically conveyed directly to the combustion unit.

Regardless of which option is finally exercised, this phase of establishing AF product form is required to complete the overall technological development of this, or any other, ultrafine coal cleaning process. Assessment of the two basic options (pelletization, CWS) are presented in the following sections.

Coal-Water Slurry Option

An intensive pursuit for research (Scheffee, 1978), development (Pommier et al, 1984; Daley, et. al. 1984), production and actual combustion testing of coal-water slurry (McHale, 1982; Perkins and Manfred, 1985; Skolnick et. al, 1981) has been conducted over the past ten years, yet there is not an appreciable market for such a product. Some utilities have even expressed reservations about testing such fuels in their boilers (Green, 1986), although large scale tests have been conducted (Perkins, 1985). Nevertheless, this form does represent a viable option for the AF product. Costs associated with dewatering the ultrafine concentrates to low levels (<20% moisture) could possibly be reduced if solids loading of CWS in the 50%-65% range is found to be acceptable. Concentrates obtained directly from filtration (35%-50% moisture) could then serve as feed for CWS production.

Yet there is some concern that use of charged surfactants in the AF process, or flocculants used in thickening process, could interfere with the stabilizing/viscosifying mechanism employed in CWS production (Turian, 1986). Another concern expressed is the use of essentially monomodal particle size distribution (AF concentrates), whereas the most efficient packing (i.e., solids loading) occurs with a bi- or multimodal particle size distribution. These factors could significantly influence the production of CWS if Aggregate Flotation concentrates are the feedstock. These areas will require laboratory investigations in order to prove that CWS is a viable product option. Such data as maximum attainable % solids loading for several different viscosifiers and stabilizers should answer concerns about chemical interferences. If there is no true interfering "charge" effect, a second option should then include studies on a coal-limestone-water slurry which have been successfully demonstrated for use in fluid-bed combustors (Heaton, et. al, 1986). AF products, both thickened and non-flocculated feeds should be considered for evaluation of rheological and stability properties with and without limestone addition.

Further efforts at evaluation of combustion characteristics may be warranted. Tests indicate that different CWS processes and chemical additive packages have a pronounced effect on viscosity and atomization (Daley, et. al, 1984). No direct correlations between atomized droplet size, which is important for good combustion performance, and viscosity at low shear (100s^{-1}) were found. Other properties, such as AF, CWS fly ash and how it relates to fouling, slagging, and tube erosion, will need to be determined.

Pelletization or Briquetting Option

Pelletization represents a viable option not only for AF product enlargement, but for fine coal recovered from waste streams. Several industries such as mineral, chemical, ceramic and pharmaceutical rely on pelletization for size enlargement, with pelletization of coal fines (28x0) being successfully demonstrated on a commercial scale (Calhoun, 1962, Charmbury, 1962, Egan, 1967; Egan and Greenwalt 1967). Economics of the process, however, are predominately determined by binder costs and the thermal drying of pellets. Yet benefits associated with pelletization of the AF product would include handling, storage and transportation of such a product.

In a recent study on pelletization of fine coal (Sastry and Fuerstenau, 1982) a number of key conclusions were reached. A summary of their findings are as follows:

- o Coals of all ranks and ash contents can be pelletized.
- o Size consist of feed coal affects pellet growth rate.
- o Binders studied all contributed to pellet strength.
- o Addition of binders affects pellet growth rate adversely.
- o Economics for the operating and maintenance costs are estimated at \$12.00/ton of coal; nearly two-thirds of this cost is accounted for by the binder and thermal drying.

A strong relationship was found between the ash and moisture contents required for fine coal pelletization. Coals with 5% ash required 43 wt% moisture, while coals with 20% ash required only 30 wt% moisture. This finding could be significant considering the fact that without post-agglomeration or thickening, ultrafine AF concentrates are most likely to have cake moistures in the 40%-45% range, with ash contents at the 3%-5% level. Pelletization of the AF product could possibly be successfully and economically produced from cakes derived from conventional dewatering devices without extensive solid/liquid separation unit operations being required. However, a recent study (Wen et. al, 1986) indicates pelletization of ultrafine coal (-400 mesh) with humic acid binders produced final products which had little resistance to impact breakage, little resistance to water disintegration, but did have good compressive strength. Pelletization may not be the ideal final product form, thus briquetting may be required.

Preliminary Combustion Characteristics of the AF Product

The University of Illinois Department of Mechanical Engineering (Drs. Buckius and Krier) recently evaluated combustion characteristics of products from the ISGS Aggregate Flotation Process (Drendel, 1987). Baseline tests were established with an ISGS tabled product and contrasted to samples from concentrates obtained during an AF pilot plant run (see Table 1.9.1). Results included analyses of exhaust gas (CO , CO_2 , SO_2 , O_2 , HC , NO_x) and flame speeds, with runs being conducted over a range of equivalence ratios. (Further efforts at injection of hydrated lime at several locations within the flame at Ca/S ratios ranging from 0 to 3 or 4 are planned.) Results indicated that the tabled and AF products burned with near equivalent flame speeds and exhibited approximately equal temperature profiles when measured at several different equivalence ratios. While both types of coal burned similarly and CO_2 , CO , O_2 and HC emissions were essentially equal, the results for SO_2 emissions indicated the positive benefits of pyritic sulfur reduction at 18.9% for the deep cleaned AF product, with calculations based on feed analysis predicting 17.1%. Additional work in this area is recommended for the next contract period if a coordinated effort can be arranged by CRSC.

Table 31. Comparison of Chemical Analyses for Preparation Plant Product and AF Product to be used in Combustion Studies¹

	<u>Preparation Plant Product</u>	<u>Aggregate Flotation Product</u>
%		
Volatile Matter	38.1	39.5
Fixed Carbon	51.5	55.1
Ash	10.4	5.4
Carbon	71.8	79.5
Hydrogen	5.0	5.8
Nitrogen	1.65	2.08
Sulfur	2.92	2.42
Sulfate Sulfur	0.367	0.123
Pyritic Sulfur	0.66	0.44
Organic Sulfur	1.89	1.86
Chlorine	0.199	0.185
BTU/lb	12856	13793
FSI	6.5	5.5

¹All values on Moisture Free Basis

RESULT AND DISCUSSION OF PETROGRAPHY AND XRD STUDIES

The primary purpose of the petrographic and mineralogic task is to support the development of the Aggregate Flotation process. Key areas of research interest have included pyrite grain size and association, coal maceral variation, mineral liberation as a function of particle size, and basic mineral characterization of feed and product samples. Work undertaken this year has included sample characterization (sampling and basic sample characteristics), evaluation of liberation data for selected samples, evaluation of products from waste slurries, and mineralogical studies. Liberation analysis results are reported separately because the focus of this work is on mineral liberation.

Sample Characterization

Efforts during the first half of the year were directed at improving old petrographic analysis procedures and developing new ones. Problems arising from analyzing pyrite in low pyritic sulfur AF samples required a more useful and robust analysis to be developed. This analysis, which measures the proportion of particles with enclosed pyrite in a random sample of particles, has initially been used to supplement information from the pyrite characterization analysis. In cases where the concentration of pyrite is low, the analysis of the proportion of particles with pyrite may be more useful than the pyrite characterization analysis used previously (Harvey and DeMaris, 1985), and could replace it in analysis of low pyritic sulfur samples. This new technique, Liberation Analysis, is discussed further in the mineral liberation section.

The data analysis programs have also been improved. Program improvements based on input from petrographers have been combined with gradual conversion of computer programs from CP/M operation to MS-DOS operation. This makes such programs execute faster and allows easier program transfer among IBM-compatible computers. This task will be completed early in the next contract year.

Due to a change in microscopes unrelated to work objectives there was a slight improvement in the resolution of pyrite grains; this now allows for a wider range of actual particle diameters. The computer program which produces grain size frequency tables is also being upgraded to handle the wider range of particle sizes.

Work is also continuing, in cooperation with R. D. Harvey (ISGS), toward adapting a simplified microlithotype analysis to the fine particle size ranges encountered in AF concentrates and wastes. The conventional microlithotype analysis was designed to handle only standard petrographically standard petrographic pellets at -28M size, and is unsuitable for very fine samples. This technique involves the use of a special reticle with five, 20 micrometer diameter circles, and evaluation of particles below 30 micrometers in diameter. Work is continuing.

The Illinois No. 2 (Colchester-W) feed sample was recollected this year because of the potential for pre-mining oxidation of the original sample, as evidenced by higher sulfate levels. This sample will be compared to the original one by additional AF batch tests to evaluate this factor. Oxidation is known to have significant influence on flotation characteristics, and may explain why this particular sample was found to be very difficult to float.

Characterization of the Illinois No. 5 (Springfield-SE) sample was done prior to any grinding study. This feed sample was roll-crushed, tabled and wet-screened; petrographic analysis was conducted on the resulting size fractions. A comparison of the pyrite cleanability index (PCI) and the high-temperature ash for this sample is shown in figure 11. (The PCI is a petrographic index of potential cleanability which has high values for uncleaned samples, and low values for relatively clean samples.) The high PCIs in the +14M fractions are due to higher levels of "pyritic coal" in those fractions, and represents the major area where the curves are distinct.

The pyrite grain distribution and size for this set are illustrated in figures 12a and 12b. Free pyrite is very low in the 14X65M fractions, but becomes significant both in proportion and diameter in the -65M fractions. The free pyrite in the +14M fractions apparently "piggybacked" on larger maceral-dominated particles during wet screening. Conversely, enclosed pyrite dominates the coarse and middle size fractions, and generally averages around 5 to 6 μm for all but the finest fractions.

Evaluation of Liberation Data

Much petrographic research effort was devoted this year to development of new analysis techniques to meet contract requirements for evaluation of very finely ground samples. A new technique Liberation Analysis was

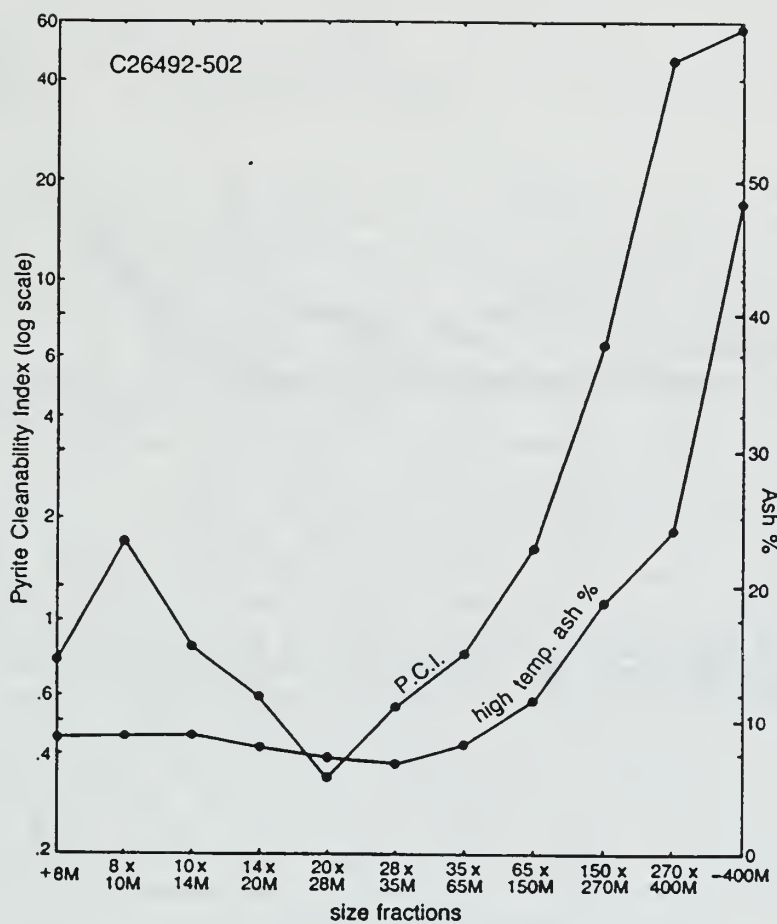
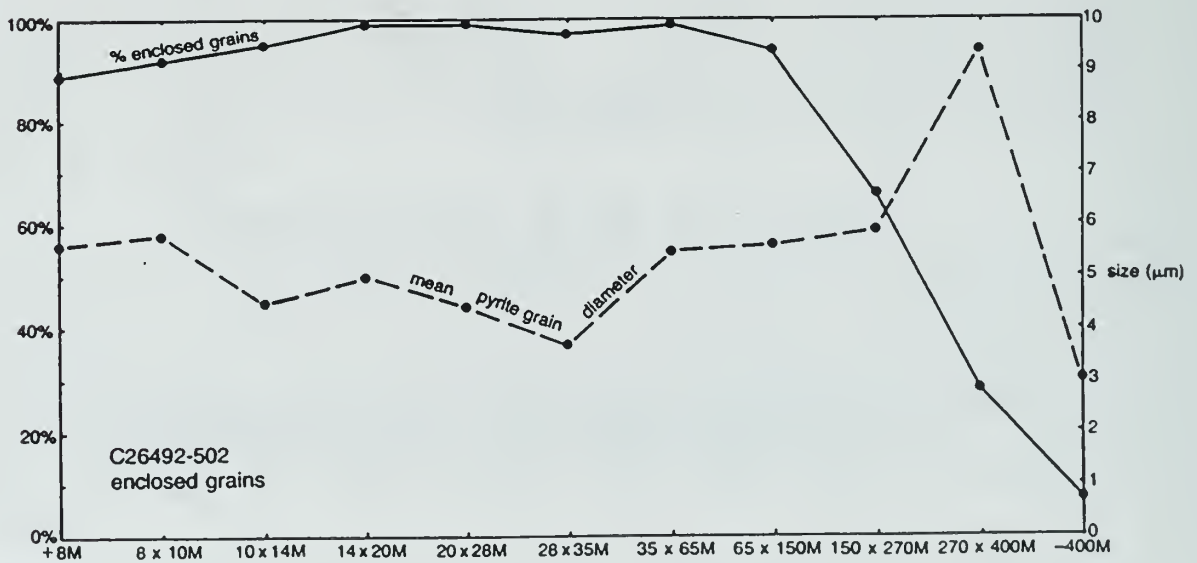
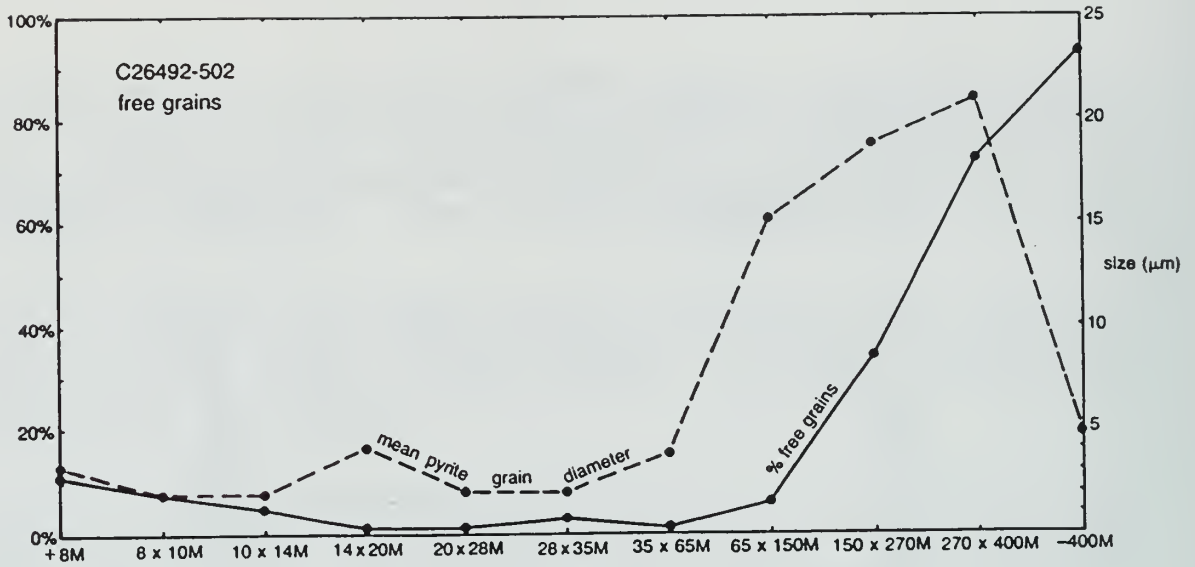


Fig. 11. Pyrite cleanability index (PCI) and percent high temperature ash for the screened Springfield-SE feed sample.



- Fig. 12.A. and 12.B. Pyrite grain diameters and percent of occurrence for free (12.A) and enclosed (12.B) grains for the screened Springfield-SE feed sample.

formalized, tested, revised and is now in use. This analysis gives direct measure of mineral presence and potential liberation after further crushing/grinding. It also provides useful data on minerals other than pyrite, an area not well addressed in the past. The operator finds 100 particles with enclosed minerals (where available), and then gathers further information on the nature and liberation potential of those minerals. For the particles with minerals the types of minerals are also recorded.

The analysis utilizes the following three questions. The first question (Q#1) evaluates whether or not minerals are present in a randomly selected set of particles. The second question (Q#2) evaluates whether or not there are visible planes of weakness in the particles containing minerals. The third question (Q#3) evaluates whether or not further breakage of those particle will likely segregate most of the mineral into one or more subparticles, leaving little or no mineral matter in other subparticles.

The advantages to the technique are that it generates direct information on all enclosed minerals, and yields useful results even when enclosed minerals are scarce. The key weakness of liberation analysis is that the cross-sectional plane through each particle which is petrographically examined is not always representative of the whole sample. All particles which have visible minerals are properly characterized, but some of those without visible minerals at the polished surface have unseen minerals. This result is an underestimation of the percent of particles with minerals which is substantial in the larger particles, +100 mesh (M), in crushed and ground samples. Many of the +100M particles examined have multiple mineral grains at the polished surface, and undoubtedly have more unseen. As the mean particle size drops the liberation analysis information becomes more reliable. Below 400M, with particles averaging under 37um across (the grid size), the underestimation is probably below 20%, since most of the remaining pyrite (by weight) is framboidal, falling in the 8-20um range. Framboids are spherical aggrerates of small pyrite crystals, often occurring in clusters. It is clear from work discussed below that substantial liberation of framboidal pyrite will require reducing much of the sample below 400M size. It is in this range that liberation analysis gives the most useful data. The evaluation of visible planes of weakness (Q.#2) and further liberation potential (Q.#3) are also improved as the particle size drops, and are much more realistic for particles passing 100M.

Mineral liberation data for representative particles from wet-screened size fractions of the Illinois No. 5 (Springfield-SE) is illustrated in Figure 13. The initial drop and plateau starting with the 28X35M fraction probably represents rather coarse pyrite agglomerates and fracture fill minerals, mostly pyrite and marcasite. A steady decline in percentage of particles with enclosed minerals below the 65x150 mesh size range, and this probably represents real liberation of the fine, widely distributed framboidal pyrite. Note that even in the 270x400M fraction, 45% of the particles with minerals had a good chance for further liberation with additional grinding (Q.#3) and 67% of those particles had visible internal planes of weakness.

Figures 14a and 14b illustrate the data available from mineral liberation work for the Illinois No. 5 (Springfield-SE) sample. Grinding times were selected to produce approximately 80 weight percent of the sample passing

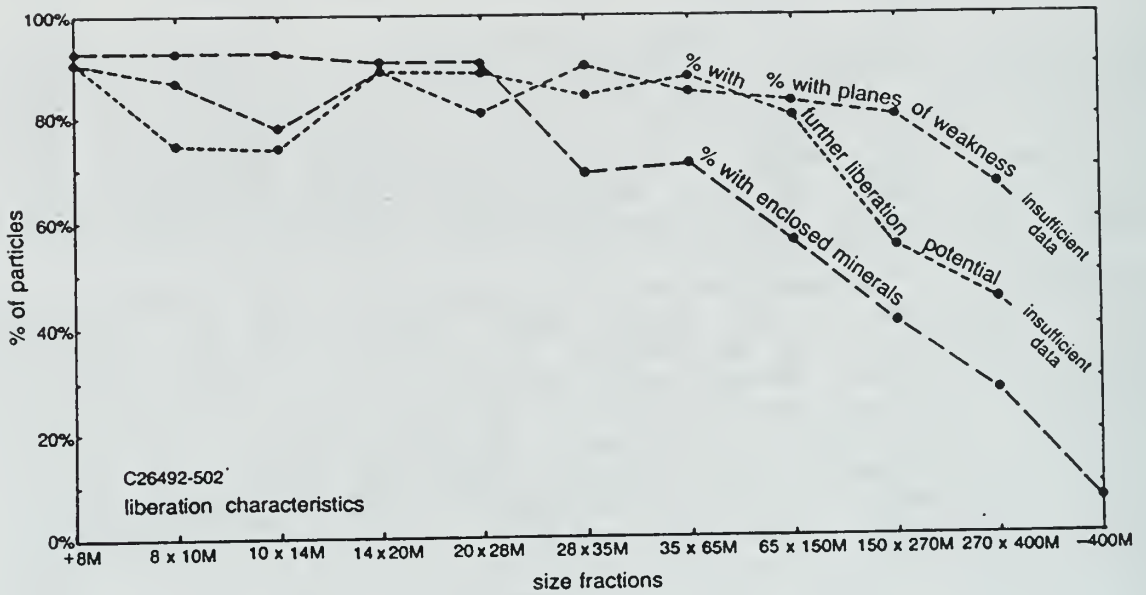


Fig. 13. Mineral liberation data for the screened Springfield-SE feed sample.

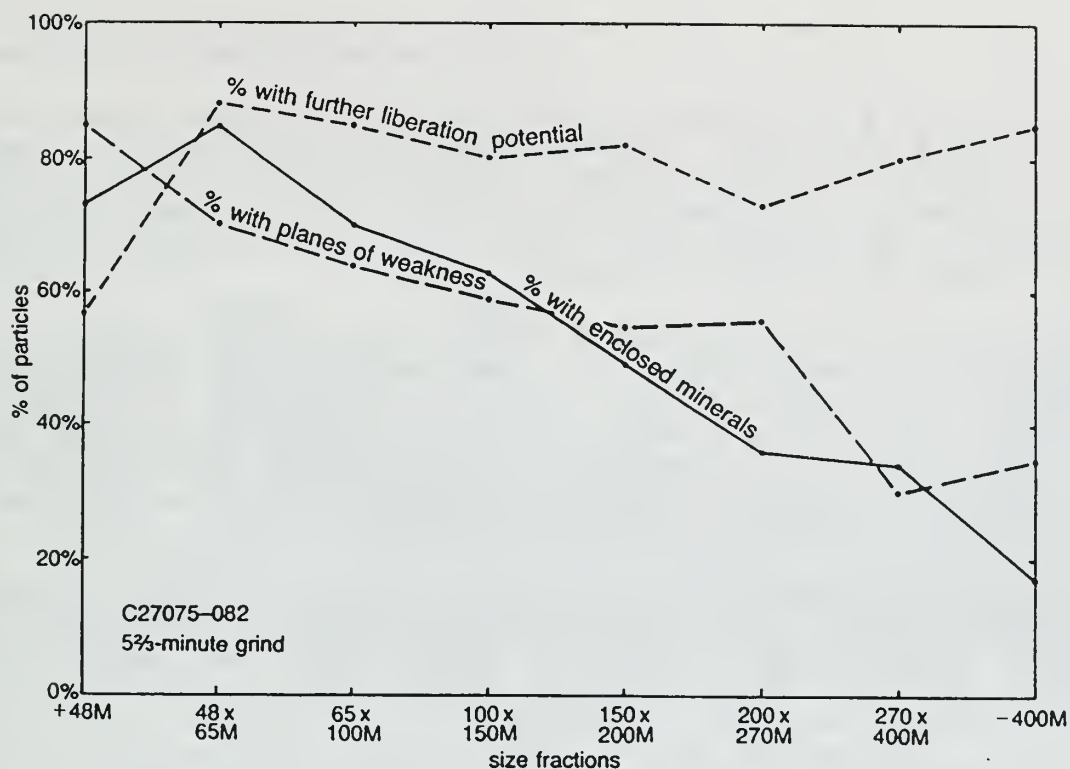


Fig. 14A. Mineral liberation data for the Springfield-SE feed following 5 2/3 minutes grinding; size consist approximate 80 weight percent passing 65M screen.

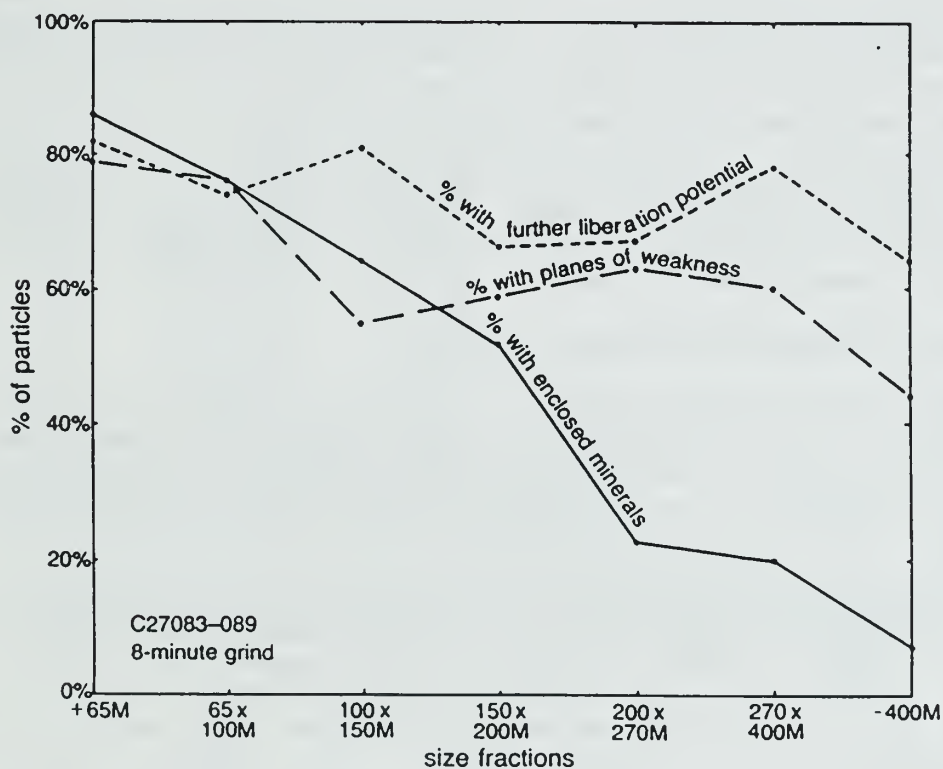


Fig. 14B. Mineral liberation data for the Springfield-SE feed following 8 minutes grinding; size consist approximates 80 weight percent passing 100M screen.

65M for the 5 2/3 min. grind, and approximately 80 weight percent passing 100M for the 8 min. grind. The enclosed mineral characteristics reported are very dependent on particle size in these two sets, and thus have very similar values for corresponding size fractions. In both cases the percent of particles with enclosed mineral grains does not fall below 50 percent until the 200X270M fraction is reached.

Comparing the 5 2/3 minute and 8 minute grinds of the Illinois No. 5 (Springfield-SE) sample (Fig. 15a) one can see most of the changes occurring in the +200M functions. Not surprisingly there is virtually no +48 material left in the 8 minute grind, and the bulk falls in the -65 to +150M range. While little very fine material is being generated, there is some suggestion in the data that the new -400M particles are more mineral-free than the original population of particles.

When the data on percent of particles with enclosed minerals is combined with weight percent of the screened fractions, a plot of calculated percent particles with enclosed minerals can be generated. The graphs are illustrated in Figure 15b. Using the weight percent data for the 5 2/3 minutes grinding set gives an average of 57% of particles with enclosed minerals. Incorporation of the weight percent data for the 8 minute grinding set gives an average of 46% of particles with enclosed minerals. Both estimates are conservative, for the reasons noted above. Because of the small mean size of the remaining pyrite, no major change in liberation occurred between these two sets.

From the above discussion it is clear that substantial liberation of the widely disseminated pyrite in the Illinois No. 5 (Springfield-SE) coal will require longer grinding times. Further grinding tests approaching 20 minutes are planned for this sample in the coming year. It appears from the preliminary data that to achieve a product where 75% of the particles have no enclosed pyrite, the size distribution will have to exceed 80% passing 270M. To reach 80% particles with no enclosed pyrite will probably require a product which is 80% passing 400M. Such higher levels of liberation will allow greater selectivity by the AF process, which should boost pyrite rejection at comparable Btu levels.

Evaluation of Products From Waste Slurries

Significant efforts were expended on evaluation of two preparation plant waste slurries. The slurries are nominally -28M products which were found to contain significant amounts of +100m material. Liberation analysis results for several screened and tabled sizes plus one AF concentrate are reported in Table 32.

Both of the waste slurry samples examined this year showed potential for further beneficiation, even though they were cleaned by AF without further grinding. Samples examined were wet-screened size fractions. The minerals in the coal from the waste slurries were not substantially different in mineral size or maceral associations than whole coal samples from elsewhere in the Illinois Basin. Thus, further grinding would increase mineral liberation, just as in other whole coal feeds examined.

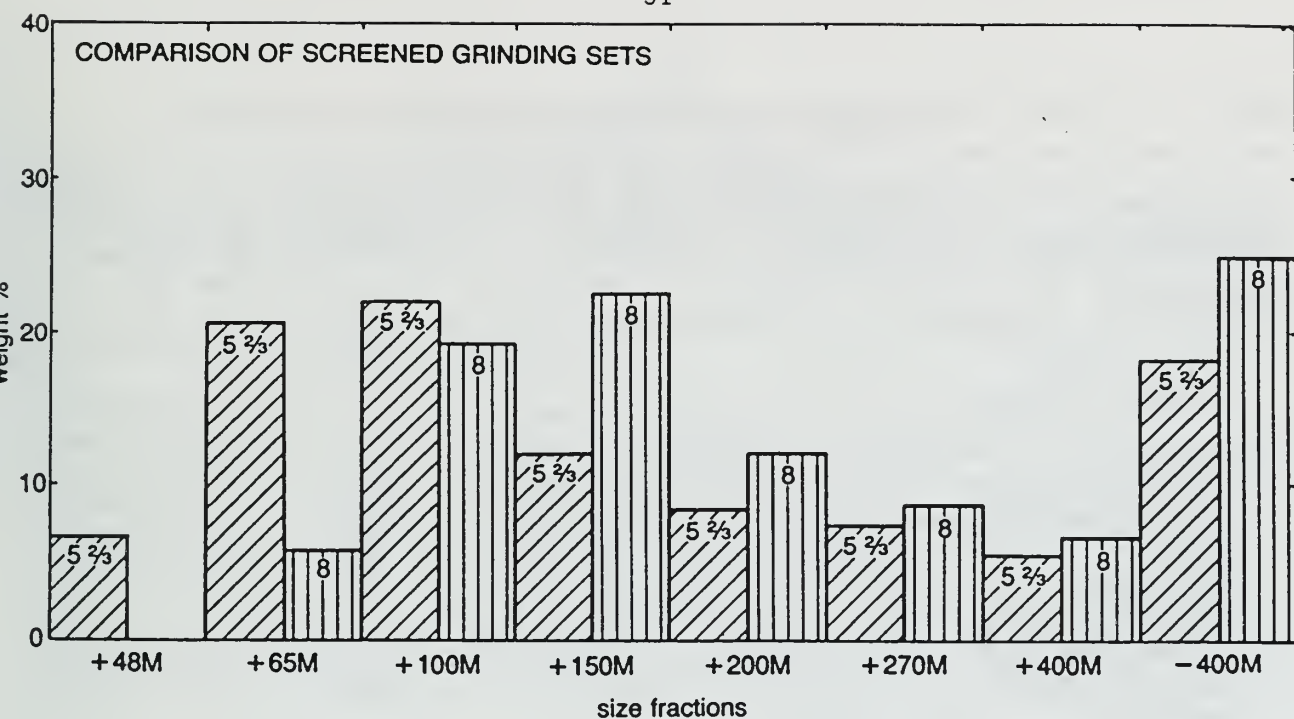


Fig. 15A. Comparison of size fractions by weight percent between 5 2/3 and 8 minute grinds.

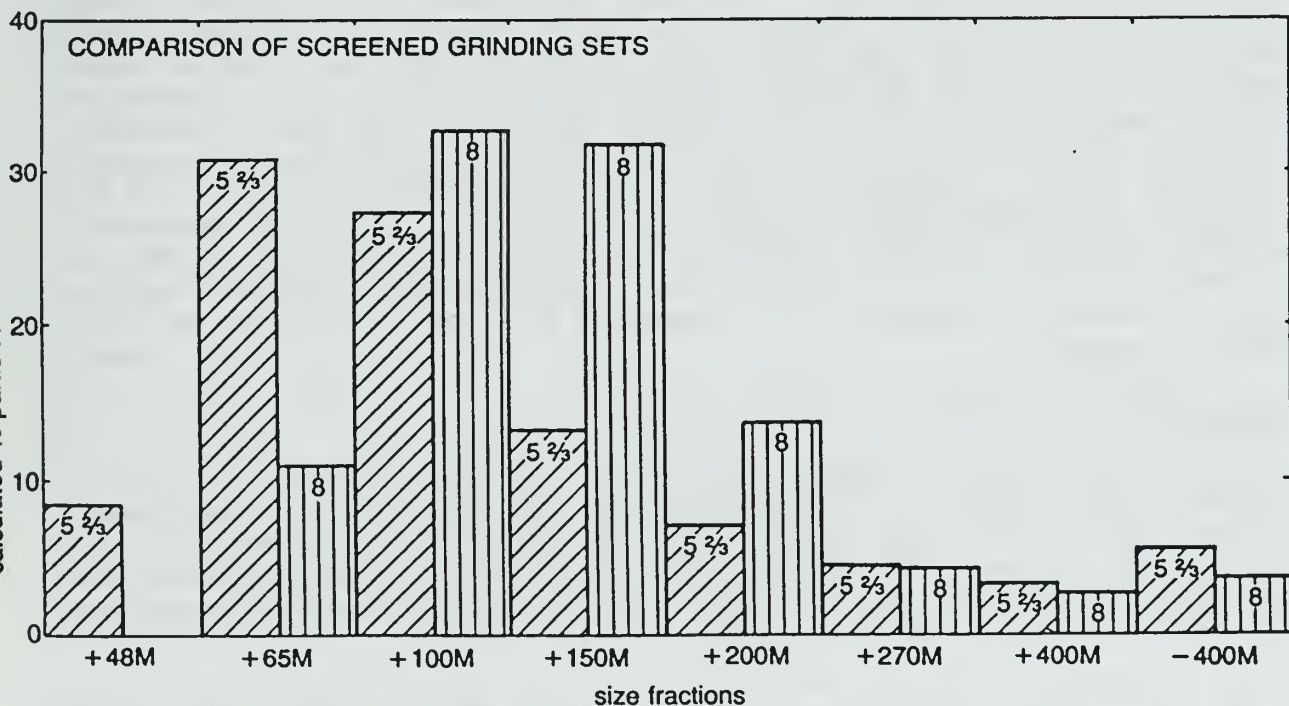


Fig. 15B. Percent of particles with enclosed minerals (petrographically derived) on a whole sample, mass-weighted basis for the 5 2/3 and 8 minute grinds.

TABLE 32. Mineral liberation data for waste slurry feed and products

Slurry code and sample type	Percent particles with:		
	enclosed minerals	planes of weakness	further liberation potential
Slurry No.1			
Dual-stage batch AF conc. from untreated refuse	26	42	70
Slurry No.2			
Untreated refuse	67	58	88
+50M fine fraction tabled	94	64	87
+50M coarse fraction, tabled	93	60	61
50X100M fine and coarse fraction tabled	78	66	76

Mineralogic Studies

Work done for the mineralogic task consisted of both X-ray diffractin (XRD) evaluation of many individual samples and improvements in the calculation of semiquantitative data. Mineral data for 47 samples were delivered over the reporting period, and this semiquantitative data was compared with chemical results for selected concentrates and tailings. A computer program was written by John Fox (Project Assistant) that gives semiquantitative weight percents for pyrite (calculated from chemical data), Kaolinite, Illite, Calcite and Quartz. Traces of other minerals, such as marcasite, are noted when observed on XRD tracks. This will make comparisons among samples in a set more accurate and convenient. Work is continuing within the clay minerals and X-ray diffraction unit to improve the match between mineralogic data generated by X-ray diffraction and full chemical analysis.

Future Petrographic Work

Plans for future work include the completion of mineral liberation evaluation for the Illinois No. 5 (Springfield-SE) coal and similar evaluation of a second coal. Petrographic evaluation of batch AF and CFU products will continue in support of AF development. Work on the ultimate cleanability of finely ground Illinois coals using float-sink tests run in a centrifuge will also be carried out.

CONCLUSIONS AND FUTURE EFFORTS

Based on the results obtained to date, ISGS Aggregate Flotation has been shown to be an extremely effective ultrafine, physical coal cleaning process. It is a technically viable approach for significantly reducing the ash and pyritic sulfur contents of Illinois Basin coals and can be utilized for a number of different feedstocks including ROM, prep plant products, waste tailings and gob.

Ultrafine grinding, which achieves liberation of ash-bearing minerals, has been integrated with the concept of "chemically selective" reagents which are specific for flotation of ultrafine coal. In this respect the need for developing or utilizing new types of cells (eg. columns, microbubble) has been reduced. Success in reagent development again, has been extremely promising. Newly developed surfactants have afforded dosage reductions by up to a factor of 7 to yield equivalent Btu recovery when compared to typical alcohol frothers such as MIBC. Additional work at ISGS has focused on preparing "mixtures" of alcohols and surfactants in attempts to attain similar results. Research and developments in this area will continue.

Characterization of products and waste by chemical, petrographic and XRD are aiding in the interpretation of the liberation/flotation phenomena. Over 400 batch AF tests involving over 5000 analytical analyses have been performed in support of this effort during the last contract year. In particular, more emphasis in the future will be placed on ash compositional studies for ROM, gravity and AF products, which may provide key information on such products when utilized as a feedstock for combustion. Areas which may lead to decreased cost or technical improvements in the process are being sought. For example, defining the overall economic limit for the fine grinding/liberation/flotation process may be accomplished by studying different particle size feed coals as a function of BTU recovery, ash and pyrite rejection. Thus one could identify the cost/benefit ratios for fine (200 mesh) versus ultrafine (400 mesh) feed. Incorporated in this effort are studies for effects of grinding on chlorine removal. Further efforts in producing low ash, low sulfur (<2% ash, 1% TS) Illinois coal by advanced physical cleaning will be attempted.

A number of an ancillary unit and operative control variables must be added to the small continuous flow unit (20-30 lbs/hr) to provide quantitative results for an accurate economic analysis of the AF process and to confirm batch AF results. Additionally, representative samples of both AF products and waste must be produced in sufficient quantity to supply other CRSC funded projects which may require such materials. Thus upgrading and retrofitting the continuous flow unit with reagent pumps, sumps, mass flow meters and equipment for consolidation of concentrates and waste streams is mandated. This next years funding will accomplish this goal.

The goal for the next year will be to continue both the fundamental and advanced development of the Aggregate Flotation process. Such issues as reagent development, economics of operation, and generating accurate and reliable data from the CFU for process evaluation, all will be undertaken. Additional aspects which are to be studied not only include reduction of pyritic sulfur, chlorine and ash, but areas such as ash composition analyses and its relationship to combustion phenomena.

Specifically, the goals for the next 12 month period are to:

- o Develop commercially feasible alcohol/surfactant mixtures which will maximize ash, pyrite rejection and BTU recovery, but further improve on dosage reduction and reagent cost.
- o Develop additional, newly synthesized collectors, and mixtures of these with alcohols, to achieve the results as described above.
- o Continue support by petrography in evaluation for liberation/ flotation characteristics of fine and ultrafine coal.
- o Determine benefits associated with ash reduction by evaluating such criteria as percentage of oxides, acid-base ratios, and slagging/fouling tendencies of feed coals and AF products.
- o Characterize surface phenomena by methods which include estimates of hydrophobicity.
- o Investigate relationships between optimum particle size/mineral matter liberation/flotation recovery in order to derive cost-benefit ratios.
- o Develop more effective circuitry design for the AF process.
- o Upgrade the continuous flow unit which will be critical if accurate data is to be made available for economic evaluation of the process.
- o Investigate chlorine reduction as a function of particle size, by advanced physical coal cleaning methods, such as AF.
- o Further test additional Illinois coals and waste streams in both batch and continuous flow tests.

ACKNOWLEDGEMENTS

The authors would like to acknowledge Dr. Chusak Chaven and his Coal Analysis Group (Ron Sjoken, Kathy Henry, Trina Kuykendall, Eltricia McMillion) for the dedicated service provided to the AF project during the past year. Also to be acknowledged are John Fox (XRD), Jim Miner (Petrography) and Steve Holtsford (AF), who provided basic support in a number of areas investigated. The authors are especially appreciative of Ms. Kim Kuchenbrod who prepared the manuscripts for all Quarterly Reports and managed to endure compilation of the Final Technical Report.

We gratefully acknowledge the Illinois Coal Development Board and the Center for Research on Sulfur in Coal for their financial support of the project.

REFERENCES

- Attia, Y. A., S. V. Krishnan, D. M. Deaton, 1983. Final Report on Selective Flocculation Technology, Group Program, Battelle Columbus Laboratories, June 30, 1983.
- Attia, Y. A., 1985. Cleaning and Desulfurization of Coal Suspensions by Selective Flocculation, in "Processing and Utilization of High Sulfur Coals," Elsevier, New York, 1985, 267.
- Bustamante, H., and L. J. Warren, 1985. Factors Affecting the Floatability of Australian Bituminous Coals, Congr. Int. Mineralurgie (15th), (2) 232.
- Calhoun, F. P., "Coal Pelletizing," Min. Cong. J., November, 1962, pp. 38-49.
- Capes, C. E., R. D. Coleman and W. L. Thayer, 1982. Selective Oil Agglomeration: An Answer to Fine Coal Treatment Problems, "Coal: Phoenix of the 80's." Proceedings 74th CIC Coal Symposium, A. M. Al-Taweel, Ed., Ottawa.
- Charmbury, H. B., "Pelletizing Process Recovers Marketable Coal from Slurry," Coal Utilization, September, 1962, pp. 19-22.
- Constantin, E., G. S. Trivett, D. Mackay, H. A. Hancock and A. M. Al Taweel, 1982. De-ashing of Low Quality Nova Scotia Coals by Spherical Agglomeration, Coal: Phoenix of the 80's, Proceedings 64th CIC Coal Symposium, A. M. Al-Taweel, Ed., Ottawa.
- Daley, R. D., G. A. Farthing and S. J. Vecchi, 1984. Coal-Water Slurry Evaluation, CS-3414, Electric power Research Institute, Palo Alto.
- Drendel, A. S., 1987. The Flammability Characteristics in Air of Mixtures of Treated Coals and Coal Derived Solids, M.S. Thesis, University of Illinois, Department of Mechanical Engineering, Urbana.
- Dumm, T. F. and R. Hogg, 1987, Washability of Ultrafine Coal, SME Annual Meeting Program, Denver, CO.
- Egan, H. N., "Pelletizing Fine Coal Recovered from Pumped Refuse," Mining Engineering, November, 1967, pp. 73-75.
- Egan, H. N. and Greenwalt, "Water Clarification, Coal Recovery and Pelletizing," AIME Annual Meeting, Los Angeles, CA, February, 1967.
- Flint, L. R., and W. J. Woarh, 1971. The Collision Efficiency of Small Particles with Spherical Air Bubbles, Chem. Eng. Sci, 26:1155.
- Fonseca, A. G., 1987. Personal Communication, January 28, 1987.
- Fuerstenau, D. W., 1980. Fine Particle Flotation, in "Fine Particles Processing," by P. Somasundaran ed., SME of AIME, NY, NY., p. 669-705.

Green, Peter, 1986. Coal Water Mixtures Await a Large Commercial Tryout, Coal Age, March, 17.

Hanson, R. D. and R. R. Klimpel, 1987. Coal Frothers, Presented at SME Meeting, Denver, Febraury 23-27.

Harvey, R. D., 1986. Personal Communication, December 15, 1986.

Harvey, R. D. and P. J. DeMaris, 1985, Size and Maceral Association of Sulfide Grains in Illinois Coals and their Washed Products: Illinois State Geological Survey Contract/Grant Report 1985-2, 49 pp. Champaign.

Heaton, H. L., E. G. Skolnik and E. T. McHale, 1986. Coal-Limestone-Water Slurries for Fluidized Bed Combustors, 8th Intl. Symposium on Coal Slurry Fuels Preparation and Utilization, Paper No. 74, May 27, Orlando.

Jameson, G. J., S. Nam and M. M. Young, 1977, Physical Factors Affecting recovery rates in flotation, Min. Sci. Engr. 9 (3) 103.

Kester, W. M. 1966. The Effect of High Intensity Magnetic Cleaning on Pulverized High Sulfur Bituminous coals, Ph.D. Thesis, Univ. of West Virginia, Morgantown, W. VA.

Liu, Y. A. 1978. Magnetic Cleaning of Coal, AMC Coal Convention, St. Louis, MO, April 24-26, 1978.

Marrucci, G., and L. Nicodemo, 1967. Coalesence of Gas Bubbles in Aqueous Solutions of Inorganic Electrolytes, Chem. Engr. Sci. 22 1257.

Mathieu, G. I. 1982. Wet and Dry High Gradient Magnetic Cleaning of Eastern Coals, in "Coal: Phoenix of the 80's," Proceedings 64th CIC Coal Symposium.

McHale, E. T. 1980 Combustion Studies of Highly Loaded Coal Slurries. DOE Cont. No. AC22-77ET13041 Phase II, Summary Report October 15, 1977-December 31, 1978.

McHale, E. T., R. S. Scheffee, N. P. Rossmeissl, T. J. Boyd and C. B. Henderson, 1982. Preparation and Burning of Coal/Water Slurry, in "Coal: Phoenix of the 80's", A.M. Al-Taweel, ed, Ottawa.

Miller, F. G. and R. M. Turbeville, 1986. Coal Flotation: Past Present and Future, SME Meeting March 2-6, 1986, New Orleans.

Miller, K. J., 1973. Flotation of Pyrite from Coal, USBM RI 7822.

Panopoulos, G. R. P. King, and A. H. Jukes, 1986. The Effect of Particle-Size Distribution on the Flotation of Two South African Coals, J. S. Afr. Inst. Min. Metall., 86(5) 141.

Perkins, R. P., and R. K. Manfred, 1985. Coal Slurry Development Program, Chem Engr. and Proc., 81 (5), 69.

Pommier, L., T. Frankiewicz and W. Weissberger, 1984. Coal Water Slurry Fuels - An Overview, Min. Met. Proc. (1)1, 62.

Read, R. B., R. R. Ruch, H. P. Ehrlinger, P. J. DeMaris, D. M. Rapp, L. R. Camp, J. A. FitzPatrick, 1986. ISGS Aggregate Flotation Fine Coal Cleaning Process, Interim Report to CRSC for the period January 1, 1983 to August 31, 1986.

Sastry, K. V. S. and D. W. Fuerstenau, 1982. Pelletization of Fine Coals. Electric Power Research Institute, Report CS-2198, Palo Alto, CA.

Schefee, R. S., 1979. Development and Evaluation of Highly Loaded Coal Slurries, DOE Cont. No. AC22-77ET13041 Phase I, Summary Report October 15, 1977-December 31, 1978.

Skolnick, E. G., E. T. McHale, H. L. Heaton, 1981. Chem of Combustion of Fuel-Water Mixtures Cont. No. N00014-80-C-0534, Office of Naval Research, Mat. Sci. Div, Arlington, VA.

Theodore, F. W., 1985. Oil Agglomeration for Fine Coal Recovery as Commercialized at Conoco-Consol. Fourth International Symposium on Oil Agglomeration, June 2-5, 1985, Toronto.

Turian, R. 1986. Personal communication, October 29.

Wen, W. W., P. D. Bergman and A. W. Deurbrouck, 1986. Aluminic Acid Binder for Pelletizing Fine Coal, Int. Coal Prep. Congress, 10th, Edmonton, Canada, September 1.

Yoon, R. H., 1984. Microbubble Flotation of Fine Coal, Dept. of Mining and Minerals Engineering, Virginia Polytechnic Institute, Blacksburg, Va., 24061, DOE grant #DE-FG22-80PC30234.

Zimmerman, R. E. 1979. Flotation of Bituminous Coals, AIME Technical Publication No. 2397, 18.

58
Appendix A

Chemical Analyses	Herrin SC1	Herrin SC2	Herrin SW*	Herrin W	Springfield SE	Colchester W
Sample Number	C23995	C24098	C22538	C24526	C23807	C23955
Ash	28.6	24.8	38.1	23.2	27.1	11.0
Carbon	56.26	61.45	46.92	58.54	55.8	nd
Hydrogen	3.39	4.12	3.28	4.17	3.89	nd
Nitrogen	1.57	1.65	0.69	1.28	1.17	nd
Sulfur	1.26	1.11	4.08	4.43	3.62	4.50
Sulfatic Sulfur	0.00	0.00	0.10	0.02	0.01	0.26
Pyritic Sulfur	0.82	0.68	2.17	2.27	2.37	3.24
Organic Sulfur	0.44	0.43	1.81	2.14	1.24	0.99
Total Chlorine	0.311	0.391	0.039	0.061	0.143	nd
BTU/pound	10,073	10,739	8,527	10,660	10,451	12,740

* IBCSP Sample 4

nd = not determined

